2008

Investigating Nickel Flux and Toxicity in Clay Sediments with Batch and Stream Recirculating Flume Experiments

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INVESTIGATING NICKEL FLUX AND TOXICITY IN CLAY SEDIMENTS WITH BATCH AND STREAM RECIRCULATING FLUME EXPERIMENTS

A thesis submitted in partial fulfillment
of the requirements for the degree of
Master of Science

By

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2008
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WRIGHT STATE UNIVERSITY

SCHOOL OF GRADUATE STUDIES

December 12, 2008

I HEREBY RECOMMEND THAT THE THESIS PREPARED UNDER MY SUPERVISION BY Christina Elizabeth Cloran ENTITLED Investigating Nickel Flux and Toxicity in Clay Sediments with Batch and Stream Recirculating Flume Experiments BE ACCEPTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS OF THE DEGREE OF Master of Science

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ABSTRACT

Cloran, Christina Elizabeth. Master of Science, Earth and Environmental Sciences, Wright State University, 2008. Investigating Nickel Flux and Toxicity in Clay Sediments with Batch and Stream Recirculating Flume Experiments.

Using batch and stream recirculating flume experiments to compare and contrast one clayey sediment (Warden Ditch) and two analytical grade clay minerals (montmorillonite and kaolinite), the dynamic interactions between two aquatic stressors (suspended solids and nickel) were explored. Aldrich humic acid was incorporated to demonstrate the mitigating effects of dissolved organic carbon (DOC) on Ni toxicity. The flux of Ni between compartments (dissolved and sorbed) was quantified as a partition/distribution coefficient. The USEPA test organism *Daphnia magna* (neonates, < 24 h) was utilized to evaluate toxicity in dynamic non-renewal, short-term bioassays. Generally, toxicity showed a linear relationship with turbidity level. Conversely, sorption coefficients were experiment specific, making them difficult to predict and assess. Clay functioned as an adsorbent, scavenging Ni. Results support the hypotheses that solids and metals act as stressors in streams, DOC attenuates the toxicity of Ni, and Ni fluxes quickly between system compartments.

CONTENTS

List of figures

List of tables

List of abbreviations and acronyms

AHA
Aldrich humic acid

ANOVA
Analysis of variance

BLM
Biotic Ligand Model

D. magna
Daphnia magna (USEPA test organism)

DO
dissolved oxygen

DOC
dissolved organic carbon

DOM
dissolved organic matter

ECxx
Median effects concentration; the concentration of a specified chemical in an exposure water that causes a nonlethal adverse effect in xx% of the organisms tested, where the effect could be immobilization, avoidance, etc.

EDTA
Ethylenediaminetetraacetic acid ($C_{10}H_{16}N_2O_8$)

FA
fulvic acid

h
hour(s)

HA
humic acid

HPW
high-purity water

HS
humic substance

HSD
Honestly Significant Difference

ICP-MS
Inductively Coupled Plasma Mass Spectrometry/Spectroscopy

K_d
Metal distribution/partition coefficient; also, sorption distribution coefficient (sediment/water distribution coefficient)

LCxx
Median lethal concentration; the concentration of a specified chemical in exposure water that causes xx% mortality

LOE
lines of evidence

M
abbreviation for a generic metal

M^{n+}
abbreviation for a generic metal cation whose charge is n^+

Ni^{2+}
The hydrated form (sometimes loosely referred to as the "free" ion) of Ni(II), where Ni(II) is nickel in its +2 oxidation state.

n
number of subjects

NOM
natural organic matter/material

NPS
non-point source

NTU
nephelometric turbidity units

POC
particulate organic carbon

SD
standard deviation

SETAC
Society of Environmental Toxicology and Chemistry

SOP
standard operating procedure

SPM
suspended particulate matter

SRF
stream recirculating flume

SS
suspended solids or suspended sediment (herein used interchangeably)

T
temperature (degrees Celsius)

TOC
total organic carbon

TSS
total suspended solids

UM
University of Michigan

USEPA
United States Environmental Protection Agency

USGS
United States Geological Survey

WD
Warden Ditch sediment

WHAM
Windermere Humic Aqueous Model; used in the BLM to calculate interactions of cationic metals and major inorganic cations with natural dissolved organic matter

WOE
weight of evidence

WSU
Wright State University

Acknowledgments

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EXTENDED ABSTRACT

Suspended solids (SS) and nickel (Ni) were manipulated as fundamental stressors in batch and stream recirculating flume (SRF) experiments to investigate flux and toxicity. Test water in each exposure chamber was characterized by measurement for temperature, pH, dissolved oxygen (DO), specific conductance (SC), and turbidity, and by analysis of samples for total, dissolved, and sorbed Ni concentration, dissolved organic carbon (DOC), total suspended solids (TSS), water hardness, and water alkalinity. Samples were withdrawn using a syringe from inside each chamber, then composited, preserved, and analyzed. The physicochemical parameters pH, temperature, water hardness, water alkalinity, dissolved oxygen, and specific conductance were similar among experiments.

A dilution series of turbid suspensions (12.5, 25, and 50 NTU) of three Ni-spiked sediment/clay minerals (Warden Ditch (WD), montmorillonite, and kaolinite) in batch experiments were sampled for total Ni, dissolved Ni, sorbed Ni, dissolved organic carbon (DOC), and total organic carbon (TOC). Acute toxicity for the 48 h exposures was evaluated by enumerating survival of the cladoceran *Daphnia magna* (< 24 h old). A Ni LC_{75} of approximately 2150 μg L^{-1} was determined. Suspensions of artificially contaminated (spiked) sediments/clays and solutions of Ni and humic acid (HA) were manipulated in various combinations (31 batch experiments and 3 SRF experiments). For batch experimental treatment, magnetic stir bars in four-liter beakers kept the solids in four replicates in suspension. To monitor toxicity for the duration of the 48 h experiment, the USEPA test organism *D. magna* was added to chambers suspended vertically in four-liter beakers. Survivals for contaminated WD, kaolinite, and montmorillonite in batch experiments were as follows: 50 NTU = 75, 65, and 23 %; 25 NTU = 90, 80, and

33 %; and 12.5 NTU = 100, 93, and 43 %, respectively. No effect on survival was observed with any uncontaminated solid at 50 NTU. As compared to the two clays, dissolved organic matter (DOM) in WD sediment had an attenuating effect on Ni toxicity. Easily desorbed Ni was released rather quickly from the SS as shown in water column Ni concentrations.

For more realistic exposures, Ni-SS at 50 NTU were introduced into the SRF in a simulated metals discharge. Experiments in the SRF also evaluated SS and Ni. Effects of one turbidity level on *D. magna* were assessed in the SRF by short-term exposures to three spiked sediment/clay types. Clay/sediment slurry was dispersed via recirculating water among organism and sample chambers placed in a randomized design. Mortality (evaluated as immobility) was assessed after 48 h. The recirculating flow attenuated the Ni toxicity as compared to batch experiments.

Along with sediment type, SS, and Ni toxicity, this research studied two sediment partitioning phases—adsorption and organic carbon. In both arenas (batch and flume), the partition/distribution coefficient (K_d) of Ni, the unique relationship between the metal and the SS, was quantified to estimate partitioning between compartments. An empirical model was used to predict the solid-liquid distribution coefficient. Perhaps due to DOM, there was a significant difference between the WD and montmorillonite treatments.

Furthermore, the research examined the influence of humic substances (HSs) on the adsorption of a transition metal to clay ligands. The role of Aldrich humic acid (AHA, a source of organic matter (OM)), defined analytically as the concentration of dissolved organic carbon (DOC), in mitigating Ni toxicity to *D. magna* was explored, as well as whether the effects of AHA (DOC) varied by sediment/clay type. As anticipated, AHA behaved as a chelator. An LC_{75} for Ni (determined from a Ni only dilution series) was combined with a dilution series of AHA to study the attenuating effects of DOC. This AHA/Ni LC_{75} dilution series demonstrated how DOC mitigates toxicity, yet only to a point, after which the AHA itself reduced *D. magna* survival. Taken as a whole, these conditions and accompanying abiotic factors constitute complex stressors [3],[4]. Results support the hypothesis that SS and Ni interact as stressors in streams and should be considered together in risk assessments.

INTRODUCTION

Aquatic ecosystems are dynamic, structurally complex, and composed of both deterministic and stochastic components. Flow is the fundamental characteristic which creates lotic systems and impacts all other parameters [5]. Contaminants such as metals are pollutants that degrade aquatic quality [6],[7]. Suspended solids are important sites for contaminant and nutrient sorption and microbial transformations [8]. Indeed, SS (rather than nutrients and metals) have been identified as the most common cause of river and stream impairment in the U.S. [9].

Suspended solids

Suspended solids and metal pollutants are ubiquitous, dominant stressors. The primary cause of stream impairment, sediment is the number one water pollutant by mass [9],[10],[11]. The U.S. Environmental Protection Agency (USEPA) identifies fluvial sediment as the single most widespread pollutant in rivers and streams, adversely affecting aquatic habitat, drinking water treatment processes, and recreation at rivers, lakes, and estuaries [12]. Sediment also is the principle conveyor of other pollutants, controlling the transfer, fate, and effect of the major contaminants (i.e., toxics) [13],[14].

Natural SS are composed of both organic and inorganic materials. Part of suspended and bedded sediments (SABS), suspended sediments/solids are the fine particles that remain in suspension due to turbulence and currents and that can be removed by filtration [10]. The suspended load typically accounts for about 90 % of the total sediment flux [15]. Because SS and bedded sediments occur naturally in water bodies and are essential to their environmental function, SABS are a unique water quality problem when compared to toxic contaminants [10]. Fine-grained sediment is an inherent and vital component of river systems and plays a major role in the hydrological, geomorphological, and ecological functioning of rivers [16]. Although sedimentation is a naturally occurring phenomenon, anthropogenic activity has caused significant changes in the quantity and quality of fine-grained sediment within lentic and lotic systems [16],[17].

Of the SS, it is the fine-grained sediments (silts and clays) that generally are associated with contaminants [18],[19]. Silts and clays, measuring less than 63 μm, are part of the wash load of streams [20],[21] and are the most easily (re)suspended particles. Specifically, clays are hydrous alumino-silicates broadly defined as those minerals that predominantly make up the colloid fraction (< 2 μm) of soils, sediments, rocks, and water [20],[21],[22],[23]. The high specific surface area, chemical and mechanical stability, layered structure, high cation exchange capacity (CEC), etc., make clays excellent adsorbent materials [24],[25]. Suspended clay is a nontoxic stressor/pollutant, but it can be lethal [26],[27]. For example, the 50 % hazardous concentrations (HC_{50}s) for suspended barite and bentonite based on the 50 % effect concentrations (EC_{50}s) were 3010 and 1830 mg L^{-1}, respectively [26].

Caused by the presence of suspended and dissolved matter such as clay, silt, microscopic organisms, organic acids, dyes, and finely divided organic and inorganic matter, turbidity (a proxy for particle concentration) is an optical property that causes light to be scattered and absorbed by particles and molecules rather than be transmitted in straight lines through a water sample [28]. In particular, turbidity is the intensity of light scattered at one or more angles to an incident beam of light, as measured by a turbidimeter or nephelometer [10]. Although turbidity is not an inherent property of water, as is pH or temperature [29], particulate optical scattering is one of the indicators used to assess the environmental health of water bodies [30]. The greater the light attenuation, the lower is the water clarity [29],[31]. Beyond aesthetic considerations, SS have direct and indirect effects on biota.

Turbidity decreases the viability of surface-water bodies [32] and damages aquatic habitat. Runoff events lead to increases in total suspended sediment levels that exceed the target water quality range [33],[32]. Sedimentation and turbidity are significant contributors to declines in populations of North American aquatic organisms [34]. Having devised a concentration-duration response model to assess the effects of toxicants, Newcombe and MacDonald [35] showed that the product of sediment concentration (mg L^{-1}) and duration of exposure (h) is a better indicator of effects than concentration alone. In their review of the literature, Newcombe and MacDonald [35] observed that the data suggest that aquatic invertebrates are at least, if not more, as sensitive to high levels of suspended sediment as salmonid fishes.

Many non-chemical and abiotic factors (e.g., SS, flow, and temperature) are a natural part of the environment, and considerable variability in their intensities and distribution occurs [3],[36],[37]. Parameters such as rainfall, flow, depth, dissolved oxygen, alkalinity, hardness, turbidity, and conductivity may fluctuate simultaneously with stressors and are useful as surrogate measures [32]. The patterns observed between water-quality conditions (such as turbidity) and survival suggest a co-varying relationship with stressors [38]. Gillis et al. [39] proved that if contaminated sediment was resuspended, there was a significant efflux of metals from the sediment into the water column, resulting in potentially toxic levels in the water column. Furthermore, fluctuating exposures of toxicants, as from nonpoint source (NPS) runoff, can produce greater cumulative effects than those that are averaged, continuous, or based on laboratory tests [40].

Nickel as contaminant/toxicant

Nickel is one of these toxicants that flux in the environment. The 24th most abundant element in the Earth's crust, Ni is relatively widespread in the environment [41],[42],[43]. Although it can exist in several different oxidation states, the prevalent oxidation state under environmental conditions is Ni(II), Ni in the +2 valence state [43]. Expressly, Ni(II) is among the prominent metal pollutants such as Pb(II), Hg(II), and Cd(II) that exert toxic and lethal effects [44],[43]. Compared to most pollutant metals, though, Ni is less toxic and more soluble. Nickel is a transition metal (a d-block element of group VIII B of the periodic table) that shows a wide range of both redox behavior and complex formation [43]. Intermediate in hardness between hard and soft metals, this divalent cation is considered a 'borderline metal' [45],[46]. Both the allergic and carcinogenic effects of Ni have been well documented [44],[43]. The USEPA water quality criteria critical maximum (Criteria Maximum Concentration, CMC) and critical continuous (Criterion Continuous Concentration, CCC) concentrations for Ni in freshwaters are 470 and 52 $\mu g\ L^{-1}$, respectively [47].

With many industrial and commercial uses, nickel and nickel compounds are ubiquitous in the environment [43],[48]. The major sources of trace metal pollution in aquatic ecosystems are discharges from domestic wastewater effluents (especially As, Cr, Cu, Mn, and Ni) and non-ferrous metal smelters (Cd, Ni, Pb, and Se). The presence of pollutant metals in the aquatic environment has caused concern because of their nonbiodegradable and toxic nature [49],[50]. Ni can be deposited in the sediment by such processes as precipitation, complexation, and adsorption on clay particles and via uptake by biota [43]. Pollutant metals such as Ni tend to sorb to clay and silt, which thereby serve as toxic contaminants. Pane et al. [51] suggested one mechanism for acute toxicity of waterborne Ni to *D. magna* is Mg^{2+} antagonism. Metal toxicity is enhanced through accumulation in living tissues and subsequent biomagnification in the food chain [52],[53],[54].

Therefore, a systematic study of the removal of Ni from wastewater is imperative for environmental health [55]. Methods such as ion exchange, solvent extraction, reverse osmosis, precipitation, and adsorption are available for treating water contaminated with toxic metals [55]. Adsorption at the metal ion-mineral interface is utilized often as a very effective way for 'scavenging' pollutant metal ions from the aqueous phase [56]. Since the adsorptive interactions in the case of low concentrations of pollutants in aqueous solution are often via ion exchange, the cation exchange capacity (CEC) of the adsorbent material is important [57]. CEC refers to the adsorption capacity for non-heavy metals (e.g., Mg^{2+}, Ca^{2+}) and relates to the sum of the CECs of the distinct chemical phases present in the sediment [58]. Like those materials mentioned by Swami and Buddhi [59], clay is a low cost sorbent of Ni and other pollutant metals [25]. For instance, Krikorian and Martin [60] extracted Ni(II), among other metal ions, from standard aqueous solutions using modified clays. Using batch systems and without adjusting the pH, better than 90 % of the metal ions could be removed [60].

Speciation

Pollutant metals such as Ni exist in various forms in nature. Familiarity with mechanisms underlying the toxicity of environmental contaminants is crucial to predicting the harmful effects of such pollutants [61],[51],[62]. Since contaminant metals occur as various species in sediments, assessing the potential toxicity of metals based on total concentrations is not sufficient [18],[42]. Speciation refers to the distribution of metal species in a particular sample or matrix [63],[64],[49]. Two pools of metals are distinguished. These groups include both naturally occurring and anthropogenically derived metals [65]. The 'exchangeable' or 'labile' pool consists of dissolved (or aqueous) species bound to DOM or colloids and those bound to sediment particles through an exchangeable binding process [66]. In contrast, the second pool consists of metals found within the mineral matrix of the sediment solids [66]. Since this 'non-labile' pool is largely unavailable to biota, only the exchangeable pool of metals is considered [66].

The exchangeable pool is susceptible to speciation in the aqueous phase and sorption to solid phases [2]. Cationic metals may bind on organic matter, (hydr)oxides, and clay minerals [67]. Sorption includes adsorption (the accumulation of matter at the solid-water interface or a two-dimensional process) and absorption (inclusion in a three-dimensional matrix) [68]. The difference between adsorption and absorption is that adsorption (relatively fast) is the attraction between the outer surface of a solid particle and a contaminant, whereas absorption (relatively slow) is the incorporation of the contaminant into the physical structure of the solid [66]. For example, adsorption of Ni(II) on acid-activated montmorillonite and kaolinite was relatively quick, with maximum adsorption observed within 180 minutes of agitation [69].

Aqueous and solid phase speciation at equilibrium is influenced by temperature, pressure, and ionic strength [58],[70]. In the aqueous phase, metals will react or bind with dissolved ligands according to the pH, Eh, ionic strength, and abundance of ligands [67],[70]. The speciation calculations of Green-Pedersen et al. [71] suggested that only two Ni species are important in the pH range of 7.0 to 8.0: Ni^{2+} is the dominant species at pH < 7.7 and $NiCO_3^0$ is dominant at pH > 7.7.

Mobility and toxicity of metals associated with sediments are affected not only by metal speciation but also granular compositions [18]. Through a sequential extraction procedure to define the following five metal speciation patterns—exchangeable, carbonate-bound, Fe/Mn oxide-bound, organic matter/sulfide-bound, and residual—Lin et al. [18] found that metal speciation in sediments had a bimodal distribution (i.e., for accumulation) over particle-size fractions (silt/clay and coarse sand). Metals in the exchangeable, carbonate-bound, and Fe/Mn oxide-bound forms were considered to be mobile and associated with anthropogenic pollution [18]. On the other hand, SS can decrease toxicity for some aquatic organisms by decreasing the free concentration of the ions [65], but at the same time they can increase the bioavailability for other aquatic organisms [72]. Thus, knowledge of metal speciation in aqueous media is important for understanding the bioavailability and mobility of Ni.

Therefore, changes in metal speciation (e.g., the free ion concentration) can dramatically affect aquatic organisms [42]. Metal ion speciation is controlled in part by NOM [18]. Through complexation, DOM may significantly affect the speciation of a number of divalent cationic metals (e.g., [73],[74],[75]). Surfaces of SS generally are coated by adsorbed organic matter [65], giving them a net negative surface charge [76],[77]. Positively charged molecules (e.g., Ni), accordingly, are attracted from the water by electrostatic forces [77],[76]. Experimental and model results indicated that the mobility, toxicity, and bioavailability of Ni in freshwater environments are determined largely by the concentration of DOM and the speciation of Ni in the aquatic system [78].

Even though Ni will complex with DOM, Doig and Liber [42] discovered that DOC concentrations (approximately 10 mg L^{-1}) representative of surface water had “little or no role in Ni speciation” under acutely toxic Ni exposure concentrations ($[Ni_{Total}]$ = 5 mg L^{-1}). In contrast, DOM “significantly affected Ni speciation” in an inverse relationship when Ni exposure concentrations were sublethal ($[Ni_{Total}]$ = 0.2 and 0.5 μg L^{-1}) [42]. Thus, speciation is critical and complex and warrants further study.

Aqueous Metal-Mineral Sorption & the Distribution Coefficient, K_d

In aqueous solutions, metal contaminants react with organic and inorganic ligands [79],[80],[2]. Reactions in which the metal is bound to the solid matrix are referred to as sorption reactions and metal that is bound to the solid is said to be sorbed [2]. Sorption is the attachment of metal species to mineral surfaces or other surfaces [67]. Adsorption to solids is one of several sediment partitioning phases for metals. Other phases include organic carbon, Fe and Mn (hydr)oxides, carbonates, and AVS. Although many physicochemical parameters are involved, the flux adsorption behavior for metals can be predicted approximately from the metal partition/distribution coefficient (K_d) [81],[82],[83],[66]. The distributing/partitioning of pollutant metals between solid and aqueous phase is quantified by a K_d [58],[84]. Thus, the flux of Ni can be monitored by determining the total, dissolved, and sorbed contaminant phases. K_d ($L\ kg^{-1}$; usually presented in log units (log K_d)), also known as the sorption coefficient, is the ratio of sorbed metal concentration (expressed in mg metal per kg of sorbing material) to the dissolved metal concentration (expressed in mg metal per L of solution) at equilibrium [67],[2]. The nominator represents the sum of the concentrations of the sorbed metal species (surface complexes) and the denominator represents the sum of the aqueous metal species (free ion and complexes) [58].

Generally,

K_d = [sorbed metal concentration, mg kg^{-1}]/[dissolved metal concentration, mg L^{-1}]

Specifically,

K_d = [sediment-Ni, μg kg^{-1}]/[water-Ni, μg L^{-1}]

The relevant sedimentary phases for trace metal sorption are the contents of organic matter, manganese (hydr)oxides, iron (hydr)oxides, and clays while the relevant aqueous phase metal complexes are those with Cl^-, DOM, HCO^{3-}, CO_3^{2-}, SO_4^{2-}, and OH^- [58],[84],[85],[86]. As gleaned from the USEPA literature search, the median Ni partition coefficient (log K_d in L kg^{-1}) for suspended matter/water was 4.6 (range 3.5-5.7, n = 30) [2].

Factors affecting sorption of metals include speciation/complexation, precipitation, colloid formation, biofixation, interactions with NOM, changes in pH, oxidation potential, salinity, competing ions, nature of sorbent phases, and surface site densities [2],[87]. Metal K_d values are not constant because solid phase composition and aqueous phase composition strongly influence metal distribution [58]. These compositions may vary considerably among water bodies and seasons. The most significant variables affecting the magnitude of K_d are pH, total concentrations of metal in solution and sorbed, nature and concentrations of important metal complexing agents (including DOC), presence of clays, weight fraction of particulate organic matter (POC) and other sorbing materials, and concentration of metal oxide binding sites (e.g., Fe and Mn) [2],[81].

Among soil, sediment, and suspended matter, there was a decreasing affinity by metals for sorption material in the order, suspended particulate matter > sediment > soil ($K_{d,\ SPM} > K_{d,\ Sediment} > K_{d,\ Soil}$) [2]. Within the three media, there also was a fairly consistent sequence in K_d magnitude for metals, partly due to characteristics unique to the metals and partly due to characteristics associated with the sorbing surfaces. Allison and Allison [2] compiled from the literature the following patterns of decreasing K_d for suspended particulate matter (based on ordering the mean K_d values from highest to lowest magnitude): Pb > Hg > Cr^{III} = Zn > Ag > Cu = Cd = Co > Ni > As.

After analysis, Allison and Allison [2] assigned values to the metal partition coefficients for soil, sediment, suspended matter, and DOC (Table 1). The method used to arrive at each assigned value (use of all or a subset of the collected literature K_d values, use of regression equations, modeling results, or expert judgment) is indicated for each metal and media-type, as is the subjectively assigned confidence level (Table 1).

Table 1. Metal Partition Coefficients (log Kd) in L kg^{-1} for Ni(II), CL[a] (1 = highest, 4 = lowest) (Adapted from Tables 3-6 of [2])

Partitioning between	Median	Mean	SD[b]	Min	Max	Comments
Soil/soil water	3.1	2.9	0.5	1.0	3.8	From literature data (raw, n=19); log-normal; CL = 1
Sediment/pore water	(no estimate)	3.9	1.8	0.3	4.0	Mean from soil Kd regression equation; (log-normal assumed); min, max from literature data; CL = 3
Suspended matter/water	4.3	4.4	0.4	3.5	5.7	From literature data (raw, n=25); log-normal; CL = 1
DOC/inorganic solution species	(no estimate)	3.7	0.9	1.9	5.4	Mean estimated from MINTEQA2 results; min, max from expert judgment; (log-normal assumed); CL = 3

[a] CL: Confidence limit
[b] SD: Standard deviation

Models for predicting K_d values differ [66]. 'Multisurface' models describe the interactions of metals with different soil constituents (e.g., organic matter, oxides, and clay) [67]. 'Empirical regression' models usually take the form of a multivariate, linear relation between log K_d and soil properties or a Freundlich type equation [87],[67]. Because they require extensive input information, multisurface models are less suited and less reliable for practical applications. Likewise, regression models may be deficient in describing the partitioning of metals in soil [67],[88]. Even so, regression models have an advantage over multisurface models because the former models usually are calibrated using a larger number of soils [67],[81]. Sauvé et al. [87] determined that K_d values were best predicted using empirical linear regressions. Generally, the simple correlation and the multiple regression analysis suggest that the ability of the soil to adsorb pollutant metals depends upon the type (mainly smectites [89]) and the amount of clay, as well as the CEC [90].

That said, field/empirical-derived K_ds are superior to model-derived K_ds [58],[81],[66],[88]. Koelmans and Radovanovic [58] created SWAMP, a detailed speciation model that simulates K_d as a function of water quality variables and estimated distribution coefficients for suspended sediments from water and sediment quality variables using empirical models. Koelmans and Radovanovic [58] found that lack of fit with field-derived K_ds was due to model flaws, deficiencies in the sensitivity/optimization procedures, and/or limitations of the analytical methodology. When the parameters were optimized empirically in a simplified version of SWAMP, simulated field K_d values were satisfactory [58]. The applicability of SWAMP for highly dynamic systems was limited because the sorption kinetics of porous solids such as natural sediments and SS may be slower [58].

On the contrary, the relatively quick, adsorptive removal of Ni(II) from water using field sediment, montmorillonite, and kaolinite is studied in this research. Removal on clay minerals, montmorillonite and kaolinite in particular, is not only feasible but proven [91],[92],[93],[56],[94],[95],[57],[96],[97],[98]. Sometimes the clay sorbents have been modified with sodium, acid, or other clays to increase their metal adsorption capacity [24],[56],[94]. Acid activation on clays enhanced their adsorption capacity compared to the untreated clay minerals by limiting possible decomposition of crystalline structure, increasing the specific surface area, increasing pore diameter, and increasing the total number of exchange sites (CEC) [69]. In general, due to a higher Langmuir monolayer capacity, montmorillonite and its modified forms had a much higher metal adsorption capacity compared to that of kaolinite or modified kaolinite [56],[24],[92],[99]. Even so, the choice of clay sorbent for the uptake of metal ions or other substances depended on the composition of the effluent to be treated [100].

Adsorption of metal by clay minerals increases with increasing pH until the metal ions are precipitated as hydroxide above pH 8.0 [56],[101],[100],[80],[68],[87]. The decreased adsorption at low pH is due to the competition between metal ions and hydrogen ions for adsorption sites on the clay surface [86]. The active sites on clay surfaces have been shown to be weakly acidic and these sites gradually are deprotonated at higher pH resulting in increased adsorption of Cu(II) and Ni(II) [91],[92],[93],[56]. Conversely, adsorption of metal ions on clay minerals decreased with increasing ionic strength [80],[71],[56],[86].

Besides pH and total metal burden (concentration), one of the most significant variables affecting the distribution of contaminants is the concentration of dissolved and particulate organic carbon [2],[87],[86]. Since the rate and extent of adsorption is critically dependent on the presence and concentration of DOM, HA was incorporated into this research to demonstrate role of organic carbon in Ni sorption and bioavailability. Martino et al. [78] found that the rate and extent of adsorption was critically dependent on the presence and concentration of DOM. The adsorption of metal ions on clay minerals decreased in the presence of organic ligands by forming stable complexes [100]. Complexation of metals with organic and inorganic ligands in solution increases the solution concentration, and therefore decreases the K_d [67].

In addition to their large specific surface area, the high CEC and the presence of both Brönsted and Lewis acidity [102],[103] enhance the adsorptive properties of clay. Employing selectivity sequence type studies, Puls and Bohn [68] examined the hard-soft Lewis acid-base (HSAB) behavior of metals and clays to explain metal-mineral sorption. The HSAB Principle states that hard Lewis acids prefer to complex or react with hard Lewis bases and soft acids prefer to complex or react with soft bases [104],[105],[106],[107],[108],[109]. Most cations are Lewis acids and most anions are Lewis bases. Hard indicates high electronegativity, low polarizability, and small ionic size, while the opposite is true for soft ions [104],[108],[109]. The hard-soft character of the clay surfaces is due to their surface functional groups. Clay minerals usually behave as soft bases relative to water, which is a very hard base [110]. The HSAB principle therefore predicts that softer cations in soil solution will tend to replace harder cations [68].

Understanding the kinetics and mechanisms of trace element sorption/desorption reactions is necessary to accurately predict potential bioavailability and risk in the environment [62],[111],[112]. Kraepiel et al. [113] determined that the sorption mechanism for montmorillonite is ion exchange. An initial rapid reaction followed by a much slower reaction was characteristic of contaminant metal sorption on clay [113],[93],[111]. The kinetics of the adsorption process was controlled by diffusion through the liquid film surrounding the solid adsorbent [91] and showed better agreement with second order kinetics [69],[92],[93],[56]. In contrast, Eick et al. [111] concluded that the reaction by kaolinite followed first order kinetics [114]. For both clay minerals, the adsorption data gave good fits with Langmuir and Freundlich isotherms [93],[67],[115].

Adsorption on montmorillonite and HA were described as linear [71]. The adsorption process was exothermic (thermodynamically favorable), with a decrease in entropy and Gibbs energy [56],[93],[69],[92].

These studies are important for assessing the long-term stability and potential availability of contaminants released into the environment [116],[111]. Interestingly, Eick et al. [111] demonstrated that desorption of transition metals such as Ni(II) adsorbed to mineral surfaces may become more intractable with time. Incorporation of this information into an exposure-effects model for benthic organisms would allow better prediction of the effect of sediment-water partitioning [117].

Organic carbon

So the magnitude and rate of sorption of Ni(II) on solids depends in part on natural organic material (NOM). Ni(II) also can sorb to NOM; in aquatic ecosystems, organic carbon is a primary partitioning phase for metals such as Ni in oxic sediments. NOM can be divided into two categories: dissolved (true dissolved matter together with colloidal material passed by a 0.45 μm membrane filter) and particulate [65]. This complex organic material is derived from decaying plants and animals and their degradation products and includes compounds containing carbon with the exception of carbon dioxide, carbonates, carbide, and metal cyanides [65]. Divided into three groups (HAs, fulvic acids (FAs), and humans), HSs are yellow-brown, high-molecular weight materials [42].

Known to associate with pollutant metals, HSs are ubiquitous components of natural waters with critical roles in mitigating metal toxicity to aquatic organisms [118],[65],[119],[120],[121],[61],[122]. DOM (quantified as DOC) reduces metal toxicity via complexation of its functional groups (carboxyl RCOOH, hydroxyl ROH, and phenolic C_6H_5OH) with metals. These functional groups (forming negatively charged anions [77]) allow HSs to chelate (bind—precipitate in some media and make solution in other media) positively charged multivalent ions, facilitating the uptake of contaminant metals via several mechanisms. Organic carbon, among other controls, thereby governs bioavailability.

Metal binding by HSs has been the focus of intensive study (see compilations by [75] and [123]). DOM is capable of "complexing metals and increasing metal solubility; altering the distribution between oxidized and reduced forms of metals; influencing the extend [*sic*] to which metals are adsorbed on suspended matter; affecting the stability of metal containing colloids; acting as metal buffers via their so-called complexation capacity; reducing metal toxicity and altering metal availability to aquatic life" [65].

In particular, DOC complexes pollutant metals, making them less bioavailable to aquatic organisms, and absorbs and attenuates harmful UVB radiation, undergoing degradation and alteration in the process [124],[86]. DOC is approximately 50 % of DOM [42]. Natural waters contain various concentrations of DOC, mostly contributed by aquatic HSs [72]. A complex mixture of large organic polymers, DOC does not bind all metals in the same manner or with the same affinity [65]. With respect to the sorption and mobility of pollutant metals, the order of bonding strength of selected ions onto DOC is $Hg^{2+} > Cu^{2+} > Pb^{2+} > Zn^{2+} > Ni^{2+} > Co^{2+}$, whereas the sequence of complexing capacity is Pb > Cu > Ni > Co > Zn > Cd > Fe > Mn > Mg [65]. The typical range of DOC in natural waters is from 1 to 15 mg L^{-1} carbon [125],[126],[127].

Factors that affect NOM complexing of metals are pH, ionic strength, NOM source characteristics, and competing ligands [65]. Probably the most important competitors in the dynamics of complexation between metal ions and NOM are the alkaline earth cations [65]. For instance, Zhou et al. [128] noted that Ni and Ca ions strongly competed for reactions with HA. Thus, high concentrations of Ca^{2+} and Mg^{2+} in contaminated water could strongly inhibit the complexation of Ni ions whereas an increase in pH and the HA concentration could attenuate such competitive interactions [128].

NOM is relevant for the immobilization and mobilization of environmental pollutants [129], thus affecting their bioavailability to biota [54],[65]. The rate and extent of adsorption was critically dependent on the presence and concentration of DOM [78]. NOM was reported to reduce silver toxicity for the highly sensitive freshwater crustacean *D. magna* [121],[122]. Thus, DOM is an important complexing organic ligand (M-DOC). The mobility of adsorbed contaminants by NOM results, as well, in transport within the natural aquatic system. During transport of metals in soils and surface water systems, metal sorption to the solid matrix results in a reduction in the dissolved concentration of metal, affecting the overall mobility and bioavailability. Concisely, NOM affects mobility, distribution, and bioavailability of contaminants in the environment.

Bioavailability

Bioavailability (i.e., "the ability of a metal to bind or traverse the cell membrane" [62]) describes the portion of a contaminant that can be taken up by the organism from its environment and food and is subsequently transported, distributed, and metabolized by the organism [62],[65],[90]. Although guidelines for metals in aquatic ecosystems are typically based on total concentration, bioavailability is crucially dependent on the speciation, or physicochemical form, of a metal. Instead of the total ‘dissolved’ metal concentration, the free (hydrated) ion activity of divalent cationic metals may be a better predictor of metal bioavailability/toxicity (see review by [63]). Bioavailability conveys the concept of the “total amount of metal in the exposure water that is accumulated and correlates well with the observed toxicity” [130],[86].

Doig and Liber [42] mentioned several factors which potentially regulate Ni bioavailability: water hardness, alkalinity, competing metal cations, competing biological ligands, competing organic and inorganic ligands (e.g., OC, Fe and Mn (hydr)oxides, carbonates), pH, salinity, and lability of metal-DOM complexes. Bioavailability of metals in sediments also is influenced by the presence of AVS [131],[132], partitioning phases, temperature, redox, and concentration of contaminant [133],[86],[134]. For instance, Ni toxicity decreased as alkalinity, hardness, and OM increased [86]. Specifically, bioavailability of metal ions decreased when water hardness increased, as a result of inorganic complexation or reduced permeability of the cell membrane [54]. Deleebeeck et al. [135] confirmed that water hardness significantly reduced Ni toxicity to cladocerans. Remarkably, the relative decrease of acute Ni toxicity to soft water organisms in ‘moderately hard’ compared to ‘soft’ test water was significantly higher than for hard water organisms in ‘hard’ compared to ‘moderately hard’ test water [135]. Furthermore, the alteration of freshwater DOC by UVB radiation exposure increased metal bioavailability [124].

The BLM was developed to explain and predict the effects of water chemistry on the acute toxicity of metals to aquatic organisms. The BLM was designed to predict metal interactions at the biotic ligand within the context of aqueous metal speciation and competitive binding of protective cations such as calcium. The biotic ligand is defined as a specific receptor within an organism where metal complexation leads to acute toxicity [134],[136],[133],[137]. Toxicity is defined as accumulation of metal at the biotic ligand at or above a critical threshold concentration [137],[138],[134],[136],[133]. While the primary mechanism of chronic toxicity seems to be respiratory impairment, the mechanism of acute toxicity of Ni to *D. magna* appears to be ionoregulatory impairment (via disruption of Mg^{2+} homeostasis rather than disruption of Ca^{2+} or Na^{+} homeostasis) [51],[86].

This modeling framework provides mechanistic explanations for the observed effects of aqueous ligands, such as natural organic matter, and water hardness on metal toxicity. The BLM incorporates the competition of the free metal ion with other naturally occurring cations (e.g., Ca^{2+}, Na^{+}, Mg^{2+}, K^{+}, H^{+}), together with complexation by abiotic ligands (e.g., DOM, chloride, carbonates, sulfide), for binding with the biotic ligand (biotic receptor), the site of toxic action on the organism [86],[139]. The sediment-BLM (sBLM) considers sediment OC, cation competition, and ligand competition. Decreased competition for binding sites between Ni^{2+} and H^{+} as pH was increased probably was a major cause of increased Ni toxicity [86]. Increasing water hardness generally decreases the toxicity of Ni to aquatic biota [140],[141]. Because of the relatively low affinity of Ni for carbonates and hydroxide, Ni toxicity probably changes little as alkalinity changes (unless pH and/or water hardness also change) [86]. The BLM constitutes an adaptable tool for predicting the acute toxicity of metals to aquatic organisms by simultaneously accounting for "metal speciation in exposure water and competitive binding of free-metal ions and other cations to biotic ligands" [86],[133],[139].

Thus, toxicity can be evaluated with test organisms. Only an organism can integrate all the factors that contribute to stress; there is no instrument or analytical approach that can measure toxicity. Easily cultured in the laboratory and sensitive to a variety of pollutants, the USEPA recommended acute toxicity test organism *D. magna* was employed for both batch and SRF experiments [142],[143]. *D. magna* is an ecologically relevant, freshwater cladoceran (microcrustacean; water flea), having a worldwide distribution in the northern hemisphere [143]. Among the most sensitive organisms to metals, *Daphnia* is commonly used as a biomonitor/bioindicator to test the toxicity of chemicals in solution or to test for water pollution [136]. Daphnids are suspension feeders, living in the water column, filtering small particles of suspended food (algae, bacteria, debris, and microorganisms), and hyper-regulating their body fluids [143],[61]. Consequently, this organism is subject to contaminant exposure via several routes of concern, including ingestion of suspended and settled sediment and DOC.

Rationale

Several concerns prompted this research. Contaminants are widespread throughout the environment. Thus far, Ni has not been studied as intensively as other contaminant metals. The ability of sediment to sequester contaminants controls many chemical and biological processes. During their comprehensive literature search, Meyer et al. [86] found no Ni toxicity tests in which only TSS concentration was varied within a study. Further, there exists a gap in the literature regarding the examination of DOM and Ni toxicity in water-only exposures. Within lotic and lentic systems, stressors (e.g., SS and Ni) impair aquatic flora and fauna. Complex stressors such as these are universal in nature and a challenge to understand [40],[4],[3],[37].

By measuring and analyzing the physical, chemical, and biological stressors of each experimental treatment, the research attempted to isolate natural and anthropogenic stressors (SS and Ni) and to predict their importance. The primary research objective was to understand the flux of SS and Ni stressors between three compartments—water, sediment, and biota—and to investigate how sediment type and level of SS affect this flux. These objectives were approached by manipulating in various combinations the type of sediment/clay, the level of SS, the arena of exposure, the concentrations of Ni, and the concentrations of AHA.

Objectives

1. Explore how Ni and SS independently and interactively affect the survival of the USEPA test organism *D. magna*
2. Determine if the independent and interactive effects of Ni and SS on *D. magna* survival are scale dependent (i.e., batch versus SRF)
3. Ascertain the magnitude of AHA (DOC) in mitigating Ni toxicity to *D. magna*
4. Verify if the effects of AHA (DOC) vary by type of SS
5. Quantify the flux of Ni between compartments—dissolved and sorbed—and determine how K_d is related to research variables

Hypotheses

- Solids and metals act as stressors in aquatic systems.
- Sediment type (i.e., clayey sediment and clay minerals) and turbidity level of SS affect the degree of toxicity of Ni to *D. magna*.
- The independent and interactive effects of Ni and SS on *D. magna* survival depend on the scale of the system.

- Both mineral and organic materials contribute to particle bound toxicity.
- DOC attenuates the toxicity of Ni to *D. magna*.
- Ni fluxes (moves from one compartment to another) between the sorbed and dissolved compartments quickly and spontaneously.
- Partitioning of Ni among the system compartments is unique to each experiment.

MATERIALS AND METHODS

Experimental sediment types and concentrations

Analytical grade clay minerals and chemicals as well as natural sediments were used in the experiments. Montmorillonite and kaolinite were purchased from Fisher Scientific and Sigma-Aldrich, respectively. Clays were used as received, without further treatment or characterization. High-purity water (HPW; deionized, ultra pure water; resistivity 18.2 MΩ•cm at 25 °C; TOC < 5 µg L^{-1}; particulates > 0.22 µm, particulates mL^{-1} < 1) produced with a Millipore Milli-Q® system (MILLIPORE MILLI-Q® Gradient water, Millipore Corporation, Billerica, MD, USA) was used for all solutions and experiments. All tests were conducted in general accordance to USEPA guidelines [144],[145],[142].

Warden Ditch

Natural clayey sediment was obtained from Warden Ditch Canal (latitude 39.868 ºN, longitude 83.961 ºW; elevation 850 feet; U.S. Geological Survey (USGS) topographic map: Yellow Springs Quad) located in Clark County, OH, USA. The nearest major town is Enon. This sediment was determined to be 75 % clay (type of clay not determined) and analyzed to have 7.7 % TOC (Table 2). Previously, the composition of WD was characterized for various pre-existing/background concentrations (Table 2) (adapted from Kevin Custer, Wright State University (WSU), Dayton, OH, USA, personal communication) [146],[147].

Table 2. Characterization and analysis of Warden Ditch (WD) sediment

ANALYSIS	UNIT	VALUE
Clay grain size	%	75
Solids	%	23.6
Total Nickel	mg kg^{-1} dry weight	18
TOC	%	7.7
Pore water DOC	mg L^{-1}	5.2
AVS	mg kg^{-1} dry weight	2540
SEM/AVS	ratio	0.00
SEM-Ni	mg kg^{-1} dry weight	6
Total Iron	mg kg^{-1} dry weight	20800
Total Manganese	mg kg^{-1} dry weight	411

Montmorillonite

Montmorillonite (Montmorillonite KSF, analytical grade, CAS number 1318-93-0, Acros Organics, Fisher Scientific, Pittsburgh, PA, USA) is a 2:1 layer clay mineral (a member of the smectite family) composed of a succession of silicon oxide, aluminum hydroxide, and silicon oxide sheets [21]. The specific surface area of montmorillonite is fives times greater than that of kaolinite. Chemically, it is hydrated sodium calcium aluminum magnesium silicate hydroxide. The molecular formula for montmorillonite is usually given as $(M^{+}_{x}{\cdot}nH_2O)\ (Al_{2-y}Mg_x)Si_4O_{10}(OH)_2$, where $M^+ = Na^+$, K^+, Mg^{2+}, or Ca^{2+} [22]. These cations are exchangeable due to their loose binding and, together with broken bonds (approximately 20 % of exchange capacity), give montmorillonite a rather high CEC (about 100 meq per 100 g), which is little affected by particle size [20]. This CEC allows the mineral to bind not only inorganic cations such as Ni but also organic cations in HA [20]. Montmorillonite has high CEC due to the substitution of the main cations with cations having lower valence [100]. Montmorillonite and its modified forms have much higher metal adsorption capacity than kaolinite [24],[56]. In addition, the water content of montmorillonite is variable; it increases greatly in volume when it absorbs water so it is described as plastic, colloidal, or swelling [24].

Kaolinite

In contrast to the relatively complex features of montmorillonite, kaolinite (Kaolinite, purum, natural, analytical grade, CAS number 1318-74-7, Fluka, Sigma-Aldrich, St. Louis, MO, USA) was selected because it is a simple type of clay and a very common, naturally-occurring colloid [21]. The structure of kaolinite (1:1 phyllosilicate) is a tetrahedral silica sheet alternating with an octahedral alumina sheet [21]. The molecular formula is $Al_2Si_2O_5(OH)_4$ [23]. Analysis has shown that there is very little substitution in the lattice [23]. In contrast to montmorillonite, kaolinite has a low shrink-swell capacity and a low cation exchange capacity (1-15 meq per 100 g) [20]. Although the CEC of kaolinite is considerably less than that of montmorillonite, the rate of the exchange reaction is rapid [23]. Kaolinite adsorbs small molecular substances. The adsorbed material can be easily removed because adsorption is limited to the surface of kaolinite (planes, edges), unlike the case for montmorillonite where the adsorbed molecules also are bound between the layers [20].

Turbidity levels of suspended solids

Turbidity levels in lotic and lentic systems vary tremendously, especially during storm events [33]. According to Griffiths and Walton [148], the upper tolerance level for suspended sediment was as low as 10-15 mg L^{-1} for benthic invertebrates. Indeed, McCabe and O'Brien [149] discerned that both filtering and assimilating rates of *Daphnia pulex* were 'severely' reduced at even low concentrations (10 NTU) of suspended silt and clay. The 24- and 48-h LC_{50} values for kaolinite toxicity to the water flea *D. pulex* were > 1.1 g L^{-1} [150]. Consequently, nominal turbidity levels for batch experiments involving solids were 12.5, 25, and 50 NTU. For comparison and contrast in a different arena, the nominal turbidity level for SRF experiments was 50 NTU.

Spiking solutions and conditions

Methods for processing and analyzing sediments were adopted from the USEPA and the USGS, as well as experts in the field. WD sediment was collected from the field using a shovel and placed into clean, acid-washed, plastic buckets, according to USEPA and USGS protocols [145] and [151], respectively). The void space within the buckets was sparged with nitrogen, after which the buckets were resealed and stored at 4 ºC in the dark until spiking [144].

Spiking involves adding one or more chemicals to sediment for either experimental or quality control purposes. Spiked sediments are used in toxicity tests to determine effects of material(s) on test species. Spiking tests can also provide information concerning chemical interactions and transformation rates.

Concentrations of spiked stock solution were calculated on the sediment dry weight basis [152]. Using wet spiking methods [145], Ni was added to sediment and clays as $NiCl_2$. Metal solutions for sediment spiking and for aqueous Ni tests were prepared from concentrated stock solutions prepared from crystalline, analytical grade Nickel(II) chloride hexahydrate ($NiCl_2.6H_2O$; f.w. 237.69; d. 3.55, CAS number 7791-20-0, Fisher Scientific, Fair Lawn, NJ, USA) and HPW (Table 3).

Table 3. Spiking with nickel chloride: one clayey sediment and two analytical clays

Name of sediment/clay	**Desired concentration of Ni [Ni] (mg kg^{-1})**	**Dry solid fraction**	**Dry weight of 1 L of sediment/clay (g)**	**Amount of $NiCl_2$ (g) per 1 L sediment/clay**
Warden Ditch	5000	0.282	333.43	6.7508
Montmorillonite	5000	n/a	963.50	19.5077
Kaolinite	5000	n/a	445.10	9.0118

Sediments were spiked at the nominal concentration of 5000 mg kg^{-1} (Table 3); actual concentrations were 5162, 4094, and 3599 mg kg^{-1} for WD sediment, montmorillonite, and kaolinite, respectively. The measured metal concentrations were within 3 %, 18 %, and 28 %, respectively, of the nominal concentration (5000 mg kg^{-1}) [153]. The spiking solutions were thoroughly mixed with the sediments in airtight, sealed 3.8 L/1 gallon Nalgene® containers [144]. The void space within the containers was sparged with nitrogen. To ensure complete and homogenous mixing, these spiked mixtures were rolled for two hours on a roller mill apparatus [152]. Containers were stored upright in the dark at 4 ºC until further mixing before use in experiments [144]. Spiked sediment/clay was equilibrated for eight weeks [144]. After equilibration and prior to the start of each test, the containers were rolled again for two hours to incorporate any interstitial water that might have separated in storage. Immediately after mixing, the spiked sediment/clay mineral randomly was added to the test container at the desired turbidity level.

For the batch experiments involving Ni solution and no sediment/clay, nickel chloride was used for the metal stock solution. The requisite concentrations for Ni only and Ni + AHA batch tests were obtained by diluting an aliquot of this concentrated stock solution. The Ni only tests comprised an irregular dilution series (nominal concentrations of 200, 375, 750, 1500, 3000, and 6000 μg L^{-1}). This wide range of Ni concentrations was selected to encompass sublethal and lethal concentrations. This essentially constituted a range-finding test that provided partial mortalities at two or more Ni concentrations [154],[144], leading to an LC_{75} for the Ni + AHA tests. Under similar research conditions, Chapman et al. [141] calculated mean LC_{50} concentrations of 1920 and 2360 μg L^{-1} for *D. magna*. To determine this lethal/toxic concentration for the Ni + AHA batch experiments, the LC_{75} for the Ni only concentration series was estimated from ToxCalc™ (v. 5.0.23, environmental toxicity data analysis software, Tidepool Scientific Software, McKinleyville, CA, USA).

Aldrich humic acid (AHA) (Aldrich humic acid sodium salt, technical grade, CAS number 68131-04-4, Sigma-Aldrich, St. Louis, MO, USA) was used as a source of DOC. The average, natural, riverine DOC level was gleaned from several sources: DOM concentrations in natural waters range from 1 to 15 mg L^{-1} carbon [125],[126],[127]. The dissolved (versus the particulate) fraction of organic matter is estimated to represent between 80 and 95 % of the TOC in most fresh waters [155],[156],[157]. Moreover, Wehr [126] observed that concentrations of DOC in rivers varied seasonally by nearly two orders of magnitude. Accordingly, a fairly wide range of AHA in was selected for batch experiments (1, 10, 25, 60, and 100 mg L^{-1}).

Common to both batch and SRF experiments

For both batch and SRF experimental water, the 50th percentile of observed water hardness in Europe (99 mg L^{-1} as $CaCO_3$) was replicated (100 ± 5 mg L^{-1} as $CaCO_3$), regarded as 'medium' in hardness. Test water was reformulated, reconstituted deionized water composed of salts dissolved in high purity, deionized water (MILLIPORE

MILLI-Q® Gradient System, Millipore Corporation, Billerica, MD, USA) (adapted from [158] and Burton Lab standard operating procedure (SOP)) (Table 4).

Table 4. Characteristics and composition of synthetic water used in all experiments

CHARACTERISTIC/ COMPONENT	UNIT	VALUE
pH	units	7.8-8.0
Specific conductance	$\mu S\ cm^{-1}$	420-440
Hardness	$mg\ L^{-1}$ as $CaCO_3$	96-105
Alkalinity	$mg\ L^{-1}$ as $CaCO_3$	90-99
$NaHCO_3$	$mg\ L^{-1}$	1920
$CaSO_4$ x $2H_2O$	$mg\ L^{-1}$	1200
$MgSO_4$	$mg\ L^{-1}$	1200
KCl	$mg\ L^{-1}$	82
$CaCl_2$	$mg\ L^{-1}$	40
NaCl	$mg\ L^{-1}$	40
NaBr	$mg\ L^{-1}$	2

Water was not renewed in either the batch or SRF experiments; the test organisms were exposed to the same solution/suspension for the duration of the 48 h tests. Experiments were performed in an open/oxic system with respect to the atmosphere but in a closed system with respect to the water. Contaminated materials were suspended in standardized conditions and a filter-feeding cladoceran was utilized as the test organism.

Organisms were obtained from the laboratory cultures of G.A. Burton, Ph.D. (WSU, Dayton, OH, USA). Bred in the lab since the 1980s, with parental organisms provided by the USEPA, *D. magna* was cultured according to USEPA protocols in reconstituted (synthetic) hard water (approximately 160-180 $mg\ L^{-1}$ as CaCO3) [142]. *D. magna* used for the laboratory toxicity testing were neonates (< 24 h old, broods two through five). Neonates were selected because the early life stages of many macroinvertebrates are more sensitive to contaminants/toxicants [159],[160]. Mortality was the acute endpoint.

Survival was determined by counting the number of living versus dead organisms (evaluated as immobility) at the end of the test.

Both the batch dynamic non-renewal acute toxicity tests and the SRF recirculating non-renewal acute toxicity tests were devised from USEPA test methods. Methods for experiments involving sediment were adapted from USEPA 600/R-99/064 [144]. Methods for batch experiments involving no sediment were adapted from USEPA 821-R-02-012 [142].

Chambers were utilized to house and expose test organisms. The chambers were cylinders constructed of transparent core tubing of cellulose acetate butyrate with a 0.067 m inner diameter, 0.070 m outer diameter, 0.0016 m wall thickness, and 0.13 m length (0.00044376 m^3 volume); polyethylene closures capped each end; two rectangular windows (0.04 m × 0.08 m) were cut opposite each other on each tube and covered with 250 μm nylon mesh (Figure 1).

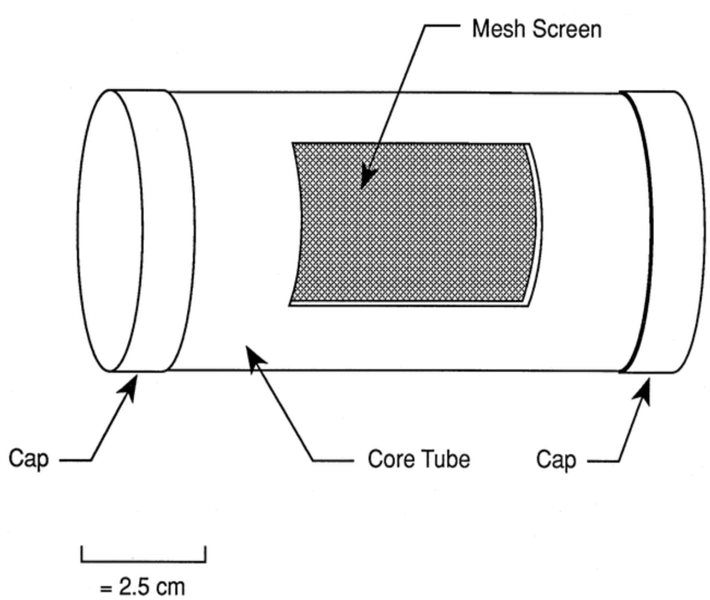

Figure 1. Chamber design. *Reprinted with permission from* [1].

Batch experimental treatment

Dynamic non-renewal, short-term batch tests were used to investigate adsorption and toxicity in the absence of the complex hydrodynamics in the flume. Also, because batch experiments required fewer resources than flume experiments, this approach provided a convenient and inexpensive way to investigate the effects of different treatments.

Batch experiments were carried out in the laboratory culture room, so they were subject to the same ambient conditions as the cultures (average temperature was 21 ± 1 ºC; photoperiod was 16 h light:8 h dark; light source was ambient fluorescent). Four controls in glass beakers placed in a water bath also were located in the culture room.

A suspension was prepared (12.5, 25, and 50 NTU) for each of three spiked solids. NTU level was determined by using a turbidimeter (DRT-15CE Portable Turbidimeter, HF Scientific, Incorporated, Fort Myers, FL, USA). Subsequent measures of the turbidity level throughout the course of the experiment indicate that the suspensions were uniform and steady.

Chambers were suspended vertically halfway down into 4 L beaker (one chamber per beaker centered at approximately the 2 L mark). Chambers were wedged between two rigid plastic rods anchored in a wooden cross bar and secured with rubber bands. To help maintain the desired turbidity level, a 250 μm mesh window was inserted into the bottom cap of each chamber. A sampling port was inserted through the top cap of each chamber (halfway down into the chamber), through which samples were withdrawn slowly with a syringe.

The batch experiments were dynamic with no renewal of the test water. Identical volumes of water (4 L) were used for all batch tests. The suspension/solution in each 4 L glass beaker was stirred magnetically with a large stir bar on a quad stir plate (speed 375 rpm; Wheaton Biostir® Magnetic Stirrer, four 4 L stirring positions, Wheaton Science Products, Millville, NJ, USA). An aliquot of spiked sediment/clays were added via syringe to stirring water in beakers.

The stressor components of the experiments were combined with one another to explore their independent and interactive effects (Appendix A). In batch tests involving solids, type of solids (WD, montmorillonite, and kaolinite) and the SS concentration (as determined by measurement of turbidity (12.5, 25, and 50 NTU)) were varied systematically to investigate different treatments. Concentrations also were varied in batch tests involving only Ni and/or AHA. Four replicates of each of the following treatments (31 tests total) were run concurrently:

- Ni only: Total concentrations of 207, 345, 750, 1516, 3079, and 6153 $\mu g\ L^{-1}$
- Sediment/clay mineral only: WD, montmorillonite, and kaolinite at 50 NTU
- SS + Ni: spiked WD, montmorillonite, and kaolinite at each of three turbidity levels (12.5, 25, and 50 NTU)
- SS + Ni + AHA: Spiked WD at 50 NTU plus AHA at 10 mg L^{-1} and spiked montmorillonite at 50 NTU plus AHA at 10 mg L^{-1}
- Ni + AHA: LC_{75} for Ni (approximately 2150 $\mu g\ L^{-1}$) at 5 concentrations of AHA (1, 10, 25, 60, and 100 mg L^{-1})
- AHA only: 5 concentrations of AHA (1, 10, 25, 60, and 100 mg L^{-1})

To quantify the fluxing Ni and to measure the amount of DOC, composite samples were collected ([Ni] at 0 h and 48 h (after equilibration), except for Ni only experiments which were sampled at time 0 h only, and DOC at 24 h), by using a syringe to withdraw aliquots of the suspension or solution from inside each of the four chambers through sampling ports inserted (extending halfway down into chamber) through the top caps. The composite Ni sample was then split into two; one was left ‘whole’ (total Ni) and one was filtered (dissolved Ni) through a pre-weighed, prepared polycarbonate filter (Isopore™ Membrane Filters, polycarbonate, hydrophilic, 0.4 μm, 47 mm, Millipore Corporation, Billerica, MA, USA). Then this filtrate and the whole sample were acidified with concentrated nitric acid (HNO_3) to pH < 2 and refrigerated at 4 ºC in the dark. Sorbed Ni was evaluated as the difference between the total and dissolved Ni samples (Appendix A). The DOC sample was collected at 24 h, filtered through a polycarbonate filter, acidified with concentrated hydrochloric acid (HCl) to pH < 2, and then refrigerated at 4 ºC in the dark (Appendices A and D). Samples also were collected for determining TSS (Appendix C).

Toxicity was tested by adding 10 *D. magna* neonates (age < 24 hours) per chamber (one chamber per 4 L beaker). Organisms were deprived of food for the duration of the experiment. Acute toxicity was evaluated by enumerating survival (evaluated as immobility) at the end of each 48 h experiment (Appendix A).

Physicochemical parameters (temperature, pH, DO, SC, and turbidity) were measured at times 0 h and 48 h with a YSI pH 100 Handheld pH or mV Instrument (serial number JC02049, YSI Incorporated, Yellow Springs, OH, USA), a YSI 85 Handheld Dissolved

Oxygen and Conductivity Instrument (serial number 02F0739 AK, YSI Incorporated, Yellow Springs, OH, USA), and the turbidimeter (Appendix D).

Stream recirculating flume (SRF) experimental treatment

To administer the SS in a more realistic way, a custom laboratory recirculating stream channel was employed which allowed the exposure of organisms to spiked solids in a flowing suspension. The SRF was located in the ‘Field Services Building’ (WSU, Dayton, OH, USA). The 2.9 m long recirculating flume (model number: S.M., serial number: Special, Living Stream (Controlled Environment for Aquatic Life) custom laboratory stream channel, Frigid Units, Incorporated, Toledo, OH, USA) was used to simulate the straight reach of a stream, with a channel 2.9 m/114 in. long x 0.3 m/11.8 in. wide x 0.3 m/11.8 in. deep (inner dimensions). The working section was fabricated of 0.019 m/0.75 in. plywood covered with 0.025 m/1 in. polyurethane insulation and fiberglass coating; a fiberglass parabolic flow form in the head of the channel (2.5 sinusoidal waves to force the flow to become fully developed in a short distance); one fixed height tailgate (Plexiglas® 0.1 m/4 in.); and two Plexiglas® windows 0.76 m/30 in. long x 0.2 m/8 in. high.

A fiberglass reservoir 0.76 m/30 in. long x 0.76 m/30 in. wide x 0.69 m/27 in. deep (inner dimensions) comprised the downstream end of the flume. Chemical resistant materials were used throughout the system. The rectangular channel and reservoir had impermeable walls and bottom and were covered with a chemically-resistant, high-solids epoxy paint. At the end of each experiment, the flume was thoroughly washed and then rinsed several times to remove the experimental sediment and recirculated water.

The flow velocity of the self-contained, recirculating channel was controlled by the operator. Water was recirculated using a centrifugal pump located downstream of the reservoir. A variable speed pump recirculated water and suspended sediment through a return pipe 0.05 m/2 in. in diameter—a circulating pump (commercially available, enclosed impeller, all stainless construction; centrifugal pump motor: U.S. Electrical Motors, Division of Emerson Electric Company, St. Louis, MO, USA; model: C511; 60 Hz; 1 HP; 1750 RPM; and an end suction centrifugal pump: Flowserve, Memphis, TN, USA; model: SFX P/M; serial number: 0404-9269; 60 Hz; 1 HP); supply piping (commercial schedule 40 polyvinyl chloride (PVC) pipe (diameter: 0.05 m/2 in.) throughout; mating flanges of PVC). Overall dimensions of SRF were 3.8 m/150 in. length; 0.89 m/35 in. width (at the reservoir); 0.74 m/29 in. height.

The standard volume of water used for all experiments in the flume was 480 L (126.8 gallons). This volume included the water in the PVC pipes but not the volume in the pump. The same hydraulic conditions were established for all flume runs. Steady, uniform flow was obtained in the channel; a pump speed of 45 Hz created a flow velocity of approximately 12 cm s^{-1} (to avoid a water temperature increase higher than 25 ºC). The velocity was measured with a calibrated, portable, water flow meter secured at various locations throughout the length of the channel. Approximately uniform flow was observed using the Flo-Mate™ Model 2000 Portable Water Flowmeter (serial number 2005058, Marsh-McBirney, Incorporated, Frederick, MD, USA). Monitoring of the current within various areas of the channel indicated that flow was evenly distributed throughout the cross section and longitude of the channel where the chambers were placed.

To the recirculating artificial stream mesocosm, a slurry of spiked natural sediment or spiked clay (i.e., WD, montmorillonite, kaolinite) was added to the head of the channel of the recirculating flume. The initial nominal turbidity level of each of the three tests was 50 NTU and was determined by using a YSI sonde (YSI 6920 V2-2, serial number 07G100772, firmware version 3.06, 6-Series multi-parameter water quality monitor, YSI Incorporated, Yellow Springs, OH, USA). Although deposition of a fine layer of clay/silt was extensive over the channel, moderate turbidity persisted for the duration of the 48 h experiments. Four exposure chambers (250 µm mesh), each containing 10 *D. magna* neonates, along with four sample/dummy chambers were placed midway along the channel in a randomized design. The replicates were secured with elastic bands to plastic-coated dish drainers; chamber caps were oriented perpendicular to flow and mesh was oriented side to side. Organisms were deprived of food for the duration of the experiment. Mortality of *D. magna* was assessed at 48 h (Appendix A).

As recommended by the USEPA, a photoperiod 16 h light:8 h dark was established [144]; a halogen work lamp provided an ambient luminance of 1000 lux. Both temperature and water level in the SRF were relatively constant. Four controls kept in beakers sitting in a water bath were monitored in the lab culture room. Via a sonde installed downstream of the test chambers at the end of channel, physicochemical parameters (temperature, pH, DO, SC, and turbidity) were continuously monitored (Appendix D).

From both the chamber and channel, Ni samples were collected at 0 h and 48 h, and DOC at 24 h (Appendices A and D). Composite samples (of four dummy chambers) were withdrawn slowly from inside the chambers via syringe through sampling ports inserted through the downstream caps of chambers. Composite grab samples were obtained near the end of the channel. The composite Ni sample was then split into two samples—one 'whole' and one filtered. Samples were acidified to pH < 2 (HNO_3 for Ni and HCl for DOC) and refrigerated at 4 ºC in the dark until analysis. To determine the concentration of TSS of each suspension, composite suspension samples (four replicates) also were collected (Appendix C).

Sample analysis

Analytical analyses of total Ni, DOC, and TOC, respectively, were performed by Dr. Chad Hammerschmidt/Katlin Bowman (WSU, Dayton, OH, USA), Brian Congiu (WSU, Dayton, OH, USA), and the Stable Isotope/Soil Biology Laboratory of the Institute of Ecology at the University of Georgia (Athens, GA, USA) (Appendix A).

Nickel

Ni samples were analyzed (in-house, Dr. Chad Hammerschmidt and Katlin Bowman) using an ICP-MS (PerkinElmer SCIEX™ ELAN® 9000 ICP-MS, PerkinElmer, Incorporated, Waltham, MA, USA) and a flame atomic absorption spectrophotometer (AAnalyst™ 400 Atomic Absorption Spectrometer, PerkinElmer, Incorporated, Waltham, MA, USA) according to USEPA methods [161]. Whole and filtered (dissolved) samples were analyzed; the difference of the two concentrations was calculated to be the sorbed fraction (Appendix A). The solid-liquid distribution coefficient (K_d) between sorbed Ni (sediment) and dissolved Ni (aqueous) (Appendix B) was calculated according to the aforementioned equation.

TOC/DOC

DOC samples were analyzed (in-house, with the assistance of Brian Congiu) using a TOC analyzer (Apollo 9000 Total Organic Carbon (TOC) Analyzer™, Combustion TOC/TN Analyzer, Teledyne Tekmar, Mason, OH, USA) and USEPA methods [161] (Appendix A). TOC of the three solids was analyzed using an elemental analyzer (NA1500 C/H/N Analyzer, Carlo Erba Strumentazione, Milan, Italy; micro-Dumas combustion assay) at the University of Georgia Institute of Ecology's Stable Isotope/Soil Biology Laboratory (Appendix A).

TSS

The concentration of TSS [28] in each suspension was quantified according to USEPA approved methods (2540 SOLIDS D. Total Suspended Solids Dried at 103-105 ºC) [162] by drying (and subsequent weighing of) 40 mL composite suspensions (four replicates) at 105 ºC (Fisher Scientific™ Isotemp™ 500 Series Economy Lab Oven, model number 516G, gravity convection oven, Fisher Scientific, Pittsburgh, PA, USA) (Appendix C).

WD grain size

Soil textural composition/particle size distribution (e.g., [147],[21],[145]) of WD sediment was determined according to the procedures described in Kettler et al. [146] (Appendix E). Silts were defined as particles finer than 62 µm in diameter and clays finer than 4 µm in diameter [153].

Statistical analysis

Statistical analyses were performed using SAS® 9 (v. 9.1.3, 2004, SAS Institute Incorporated, Cary, NC, USA) and Microsoft® Office Excel® 2007 with the assistance of Beverly Grunden of the WSU Statistical Consulting Center. Statistical analyses involved one- and two-way ANOVAs, including two-way factorial ANOVAs, Friedman's test, and multiple linear regressions (Appendix F). All model assumptions were tested and satisfied by the method that ultimately was used. A level of significance of $\alpha = 0.05$ was used in all analyses. All calculations were based on measured Ni concentrations. In the few cases when no data was available, a method of imputation was employed to artificially create an estimate of what that observation might have been [163]. The Ni LC_{75} was determined via ToxCalc™ (v. 5.0.23, Tidepool Scientific Software, McKinleyville, CA).

Survival of the test organism was calculated as the mean percent plus or minus the actual standard deviation. Likewise, physicochemical parameters were compiled as the mean ± SD. Moreover, the distribution coefficient was calculated from the measured Ni concentrations at 48 h (so that equilibrium was assured).

RESULTS

Except for turbidity, which was intentionally varied, physicochemical characteristics of all batch and SRF experiments were similar (Table 5 and Appendix D). Small differences were attributed to slightly different ambient conditions. Therefore, effects from pH, temperature, water hardness, and alkalinity were minimized. All measured water quality parameters would not be expected to have deleterious effects on the test species. Additionally, since HAs (such as AHA) and FAs contain on average 50 % carbon [42], measured DOC values also were reasonable (Appendices A and D).

Table 5. Mean physicochemical parameters for 31 batch and 3 SRF experiments

MEAN PHYSICOCHEMICAL PARAMETERS FOR 31 BATCH & 3 SRF EXPERIMENTS						
Statistic	pH (units)	Temperature (°C)	DO[a] (mg L^{-1})	Specific conductance (µS cm^{-1})	Hardness (mg L^{-1} as $CaCO_3$)	Alkalinity (mg L^{-1} as $CaCO_3$)
Batch mean	7.89	21.5	7.87	440	100	94
SRF mean	8.23	22.2	8.98	534	102	96
Grand mean	7.92	21.5	7.97	449	100	94

[a] DO: Dissolved oxygen

In contrast to these similar physicochemical parameters, turbidity of the three clays intentionally was adjusted to 12.5, 25, or 50 NTU. Besides the pure analytical clays (montmorillonite and kaolinite), the WD sediment was characterized as 75 % clay (Appendix E). Due to the small size of the clay particles, the clay material easily was kept in suspension via stir/flow and by movement of the swimming daphnids. Contrary to the adjusted turbidity levels, the sediments were contaminated at one nominal concentration. Actual Ni concentrations of spiked sediment/clay (5162 mg kg^{-1} for WD; 4094 mg kg^{-1} for montmorillonite; 3599 mg kg^{-1} for kaolinite) were not significantly different (p-value = 0.2612) from the nominal concentration (5000 mg kg $^{-1}$). Regardless of the outcome of the experimental treatments, survival of control organisms for all experiments was 100 %.

Summary graphs for batch and SRF experiments

In response to actual Ni concentrations of 207, 345, 750, 1516, 3079, and 6153 µg L^{-1} in Ni only batch experiments, survival of *D. magna* was 100 %, 83 %, 65 %, 53 %, 15 %, and 0 %, respectively (Figure 2; Appendix A).

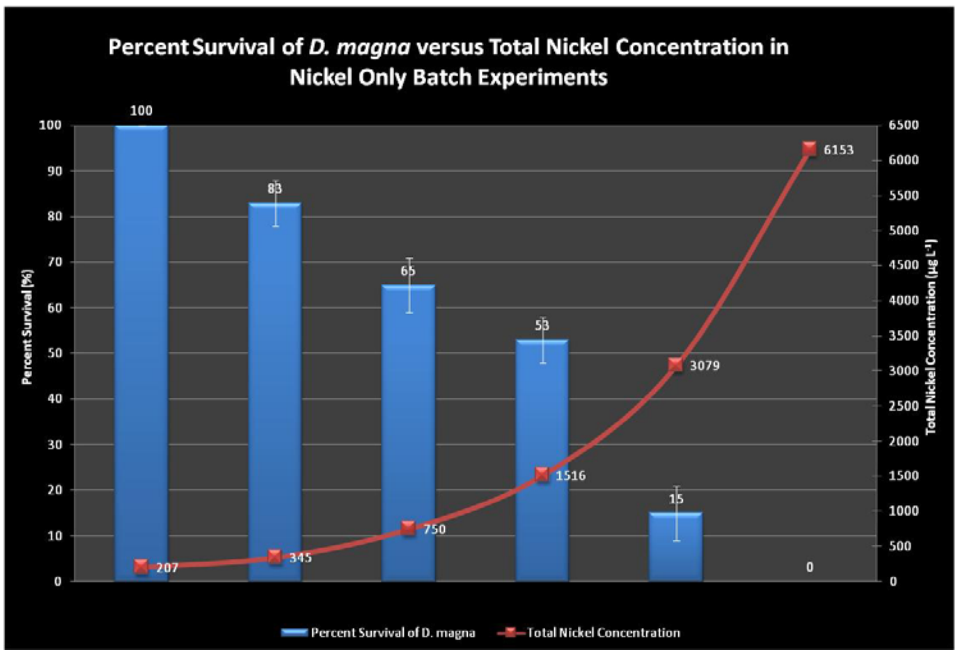

Figure 2. Percent survival of *D. magna* versus total nickel concentration in nickel only batch experiments

On the contrary, no observed effect on survival was observed with any uncontaminated solid at 50 NTU in sediment/clay only batch experiments within the 48 h exposure time (100 % survival) (Figure 3; Appendix A).

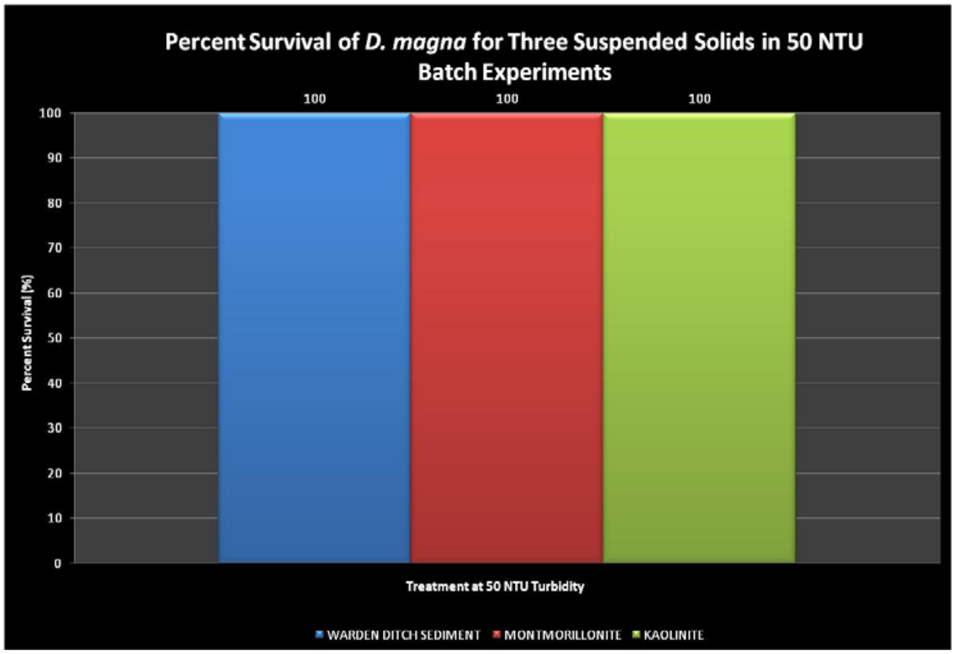

Figure 3. Percent survival of *D. magna* for three suspended solids in 50 NTU batch experiments

Within 48 hours in batch experiments, higher test concentrations of kaolinite (50 NTU) SS caused 35 % mortality while lower concentrations (12.5 NTU) enabled 93 % survival. Likewise, higher test concentrations of WD caused 25 % mortality while lower concentrations enabled 100 % survival. In contrast to both WD and kaolinite, montmorillonite at 50 NTU resulted in 77 % mortality, while at 12.5 NTU allowed only 43 % survival. Survival rates at the intermediate turbidity level of 25 NTU were 90 % for WD, 80 % for kaolinite, and 33 % for montmorillonite (Figures 4 and 5; Appendix A).

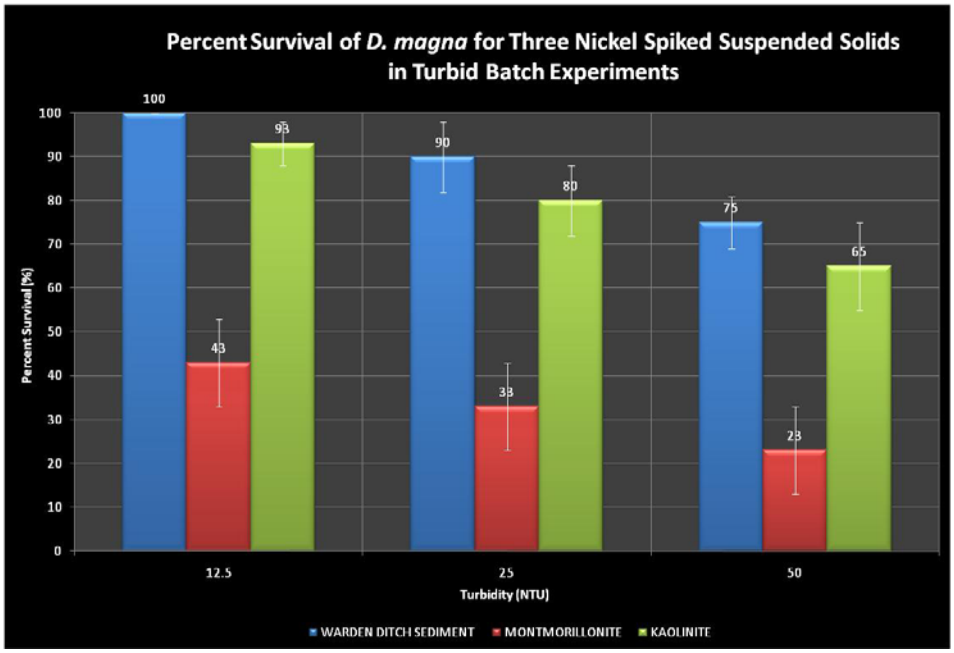

Figure 4. Percent survival of *D. magna* for three nickel spiked suspended solids in turbid batch experiments

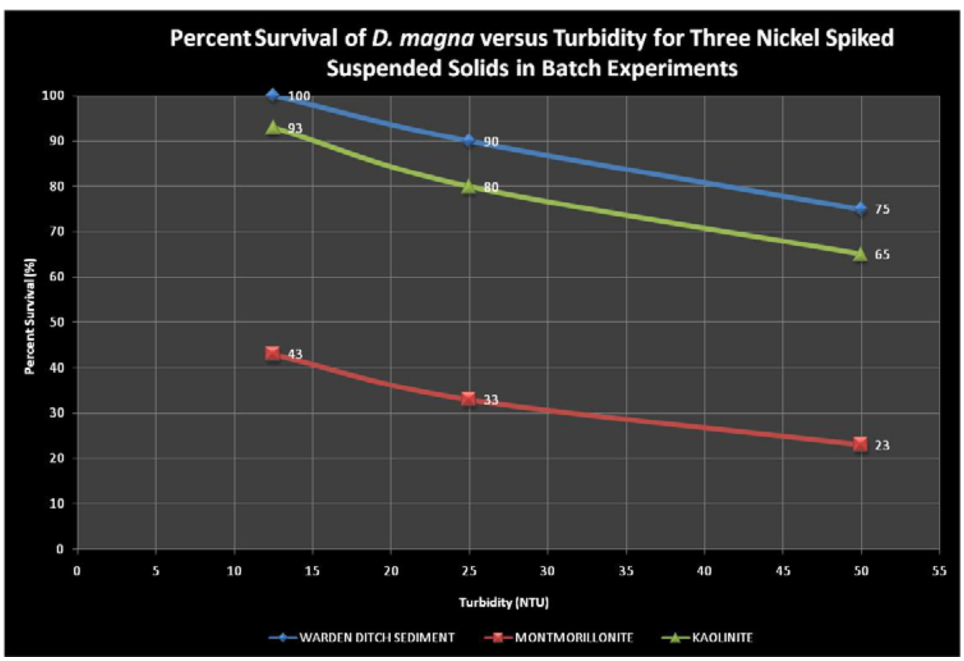

Figure 5. Percent survival of *D. magna* versus turbidity for three nickel spiked suspended solids in batch experiments

Next, the two experimental arenas were compared. Percent survival at 50 NTU in the SRF was higher than percent survival at 50 NTU in batch for each sediment/clay: 85 %, 73 %, and 33 % in the SRF as compared to 75 %, 65 %, and 23 % in batch for WD, kaolinite, and montmorillonite, respectively (Figure 6; Appendix A).

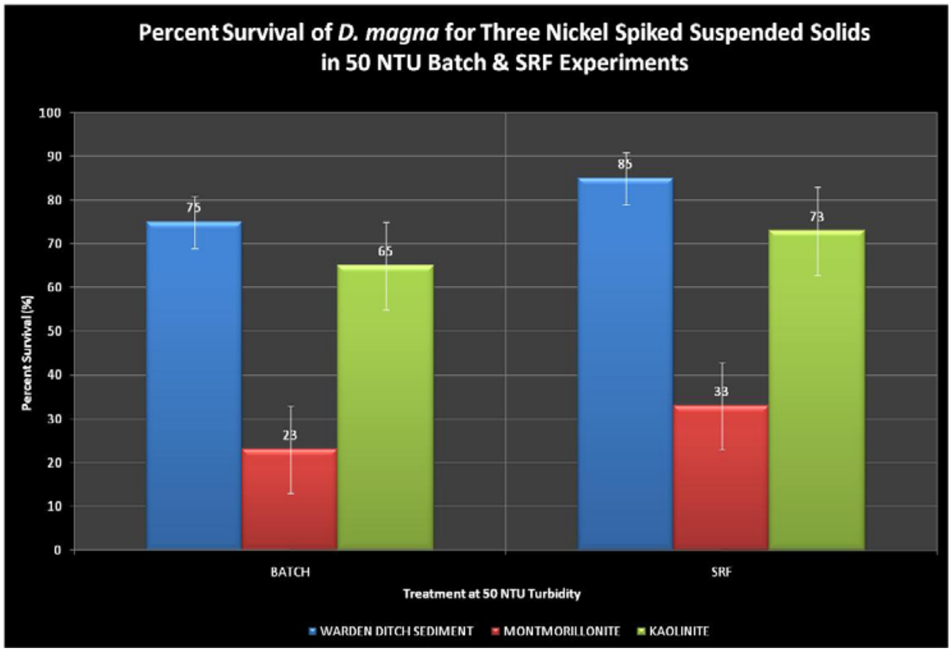

Figure 6. Percent survival of *D. magna* for three nickel spiked suspended solids in 50 NTU batch and SRF experiments

Yet another component was introduced to determine whether HA would mitigate the toxicity of Ni to *D. magna*, as anticipated. When 10 mg L^{-1} AHA was added to 50 NTU suspensions of spiked WD and montmorillonite in batch experiments, percent survival increased (93 % and 50 %, respectively) as compared to 50 NTU suspensions without added AHA (75 % and 23 %, respectively (Figure 7; Appendix A).

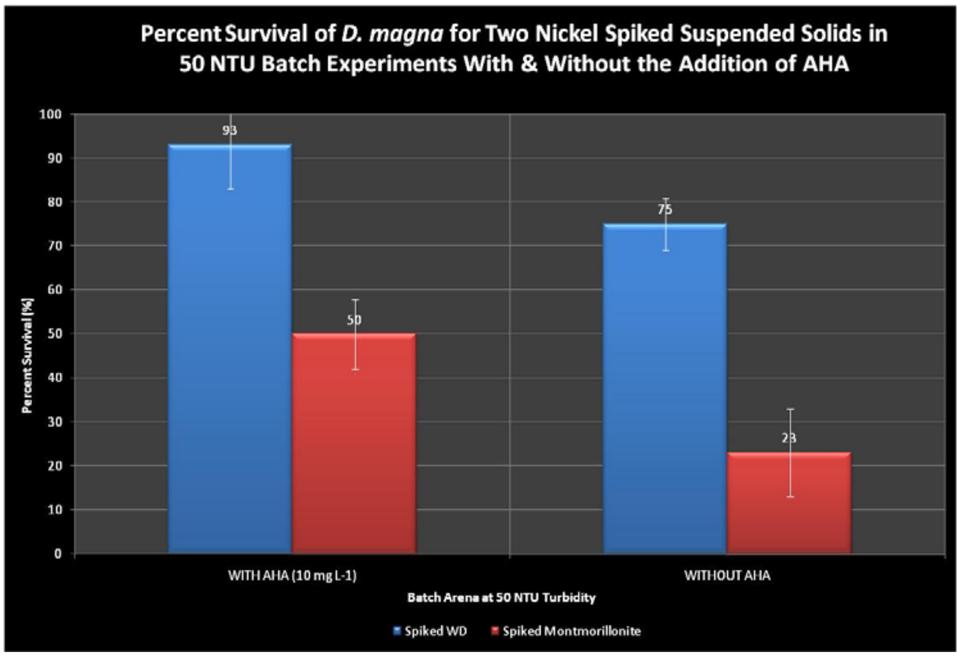

Figure 7. Percent survival of *D. magna* for two nickel spiked suspended solids in 50 NTU batch experiments with and without the addition of AHA

Absent any sediment, increasing amounts of AHA (0, 1, 10, 25, 60, and 100 mg L^{-1}) added to the same concentration of dissolved Ni (LC_{75} = 2150 µg L^{-1}) enabled greater survival up to a point, after which survival decreased—25 %, 25 %, 28 %, 43 %, 30 %, and 15 %, respectively (Figure 8; Appendix A).

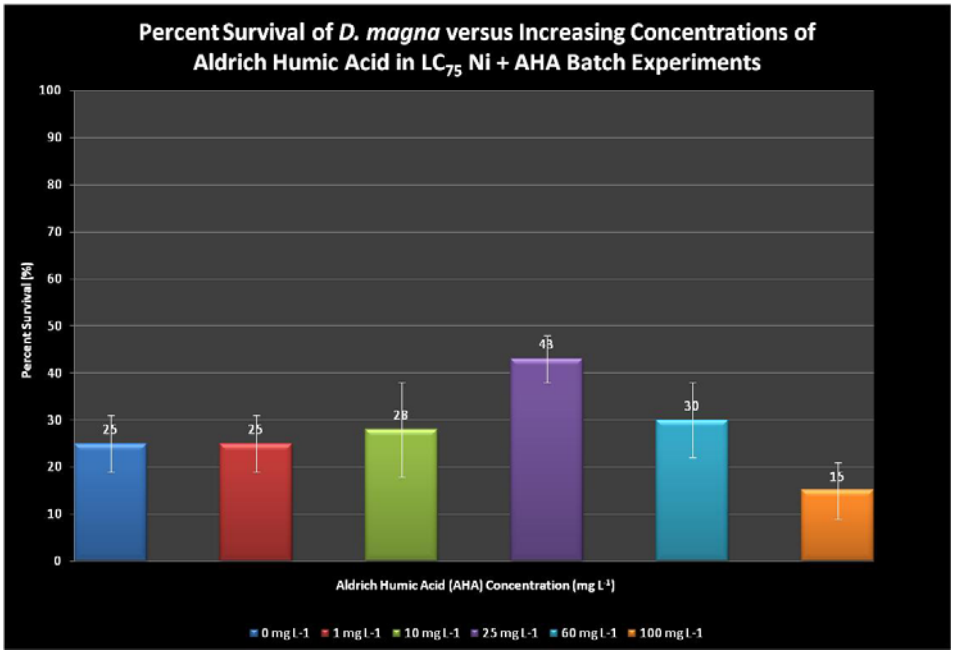

Figure 8. Percent survival of *D. magna* versus increasing concentrations of Aldrich humic acid in LC_{75} Ni plus AHA batch experiments

To verify that AHA itself was causing decreased survival of *D. magna* in these higher AHA concentrations, AHA was tested unaccompanied by SS or Ni in batch experiments. Without the presence of Ni or sediment, increasing concentrations of AHA alone (1, 10, 25, 60, and 100 mg L^{-1}) decreased survival of the test organism—100 %, 100 %, 95 %, 85 %, and 45 %, respectively (Figure 9; Appendix A).

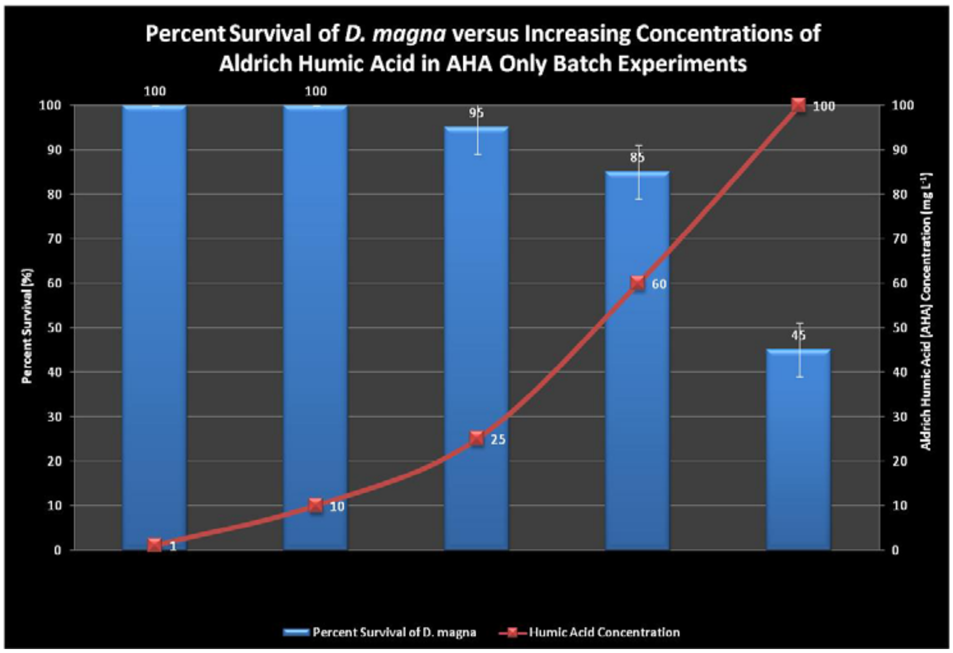

Figure 9. Percent survival of *D. magna* versus increasing concentrations of Aldrich humic acid in AHA only batch experiments

Statistical results

ANOVAs were used to explore how Ni and SS independently and interactively affected the survival of *D. magna*. The mean survival rates among all Ni concentrations in Ni only batch experiments were not the same (p-value < 0.0001); each mean was significantly different from all the others. Post hoc tests using Tukey's Honestly Significant Difference (HSD) indicated that each mean was significantly different from all the others. A box plot supports this result (Appendix F, Figure 1).

Likewise, the mean survival rates among SS with and without the presence of Ni were not the same (p-value < 0.0001). The interaction between Ni and SS was significant (p-value < 0.0001), indicating that the differences between the presence and absence of Ni were not the same for all SS. In other words, the differences among the SS in the absence of Ni were not the same as in the presence of Ni. Specifically, the survival rate dropped significantly in all the SS when Ni was introduced; survival of *D. magna* decreased as the turbidity level of the spiked sediment increased. However, in montmorillonite, survival dropped to a level that was significantly lower than in WD and kaolinite (Appendix F, Figure 2).

Similarly, the mean survival rates for all clay sediments at different levels of turbidity (12.5, 25, and 50 NTU) were not the same (p-value < 0.0001). The interaction between NTU and SS was not significant (p-value = 0.8980), indicating that any effects for NTU should be the same across all levels of SS and vice versa. The mean survival rates were not the same for all SS (p-value < 0.0001). Post hoc tests using Tukey's HSD indicated that the mean survival rates were all significantly different from each other (Appendix F, Figure 3). Equally, the mean survival rates were not the same for all turbidities (p-value < 0.0001). As for SS, post hoc tests using Tukey's HSD indicated that the mean survival rates were all significantly different from each other (Appendix F, Figure 4). Even so, there was insufficient evidence in the data to conclude that the mean log K_ds among all cells of the two-way combinations of NTU and SS were different (p-value = 0.2255) [163].

Again, ANOVAs were employed to explore how survival rates and log K_ds were affected by SS (WD, montmorillonite and kaolinite) and scale (batch, SRF chamber, and SRF channel). The mean survival rates among all cells of the two-way combinations of SS and scale were not the same (p-value < 0.0001); that is, the mean survival rates were different for all SS at different levels of scale. In particular, the interaction between SS and scale was not significant (p-value = 0.9971), indicating that any effects for SS should be the same across all levels of scale and vice versa. Nor were the mean survival rates the same for all SS (p-value < 0.0001). In fact, post hoc tests using Tukey's HSD indicated that the mean survival rates were all significantly different from each other. To be precise, lower mortality for *D. magna* occurred at the same spiked sediment level in the SRF as compared to batch treatment. The mean survival rates were not the same for all levels of scale (p-value = 0.0109). Post hoc tests using Tukey's HSD actually indicated that the mean survival rates for both SRF chamber and SRF channel were significantly different from that for batch, but there was not enough evidence to conclude that SRF chamber and SRF channel were significantly different from each other.

With respect to the partition coefficient, the mean log K_ds among all cells of the two-way combinations of SS and scale were not the same (p-value = 0.0073); that is, the log K_ds were different for all SS at different levels of scale (batch, SRF chamber, and SRF channel). The mean log K_ds were not the same for all SS (p-value = 0.0230). Explicitly, post hoc tests using Tukey's HSD indicated that the mean log K_ds for WD and montmorillonite were each significantly different from those for kaolinite, but we cannot conclude that they were significantly different from each other. Furthermore, the mean log K_ds were not the same for all levels of scale (p-value = 0.0049). Post hoc tests using Tukey's HSD indicated that the mean survival rates for batch and SRF channel were significantly different from those for SRF chamber. However, there was not enough evidence to conclude that batch and SRF channel were significantly different. Thus, montmorillonite had more in common with WD than with kaolinite; alternately, WD behaved more like montmorillonite than kaolinite. As regards survival rates and log K_ds, arena differences between batch/SRF channel and SRF chamber experiments were apparent for SS and scale.

Together with ANOVAs, scatter plots and interaction (means) plots were utilized to ascertain the extent of AHA in mitigating Ni toxicity to *D. magna*. From ToxCalc™ software, the Ni LC_{75} for *D. magna* in batch conditions was determined to be approximately 2150 μg L^{-1}. When Ni was present (at the LC_{75}), the mean survival rates were different among different levels of AHA; namely, the mean survival rates among all levels of AHA were not the same (p-value = 0.0010). Yet again, post hoc tests were run using Tukey's HSD. The largest mean survival rate occurred at AHA = 25 mg L^{-1} and was significantly larger than the mean survival rates for 0, 1, and 100 mg L^{-1} of AHA. Other paired comparisons did not indicate significant differences. Box plots of the survival distributions support this result (Appendix F, Figure 5).

Moreover, the mean survival rates were different among different levels of AHA with and without the presence of Ni. The mean survival rates among all levels of AHA and Ni were not the same (p-value < 0.0001). In this case, the interaction between AHA and Ni was significant (p-value < 0.0001), indicating that the effects of AHA in the presence of Ni may not be the same as the effects in the absence of Ni (Appendix F, Figure 6 and Table 9).

True to form, the scatter plot was very similar to the interaction plot (Appendix F, Figure 7). In the presence of Ni, 25 mg L^{-1} of AHA yielded the highest mean survival rate, but it was not significantly different from the mean survival rate at 60 mg L^{-1}. Hence, contrary to what one may presume from examining the survival data, the 'critical value' does not necessarily occur between 25 and 60 mg L^{-1}. Addition of AHA to the LC_{75} Ni solution attenuated the toxicity of this B type/borderline metal up to a point, after which the concentration of AHA itself actually decreased survival of *D. magna*.

To explore how survival rates and log K_ds were affected by two SS (WD and montmorillonite) and the presence or absence of AHA, two-way ANOVAs once more were selected. The mean survival rates among all cells of the two-way combinations of SS and presence or absence of AHA were not the same (p-value < 0.0001); that is, the mean survival rates were different for SS in the presence and absence of AHA. Even so, the interaction between SS and AHA was not significant (p-value = 0.2577), indicating that any effects for SS should be the same across all levels of AHA and vice versa. The mean survival rates were not the same for all levels of AHA (p-value = 0.0002). Post hoc tests using Tukey's HSD indicated that, regardless of the type of SS, the mean survival rate when AHA was present was significantly larger than when AHA was absent. The mean survival rates were not the same for all levels of SS (p-value < 0.0001). Post hoc tests using Tukey's HSD indicated that, regardless of the presence or absence of AHA, the mean survival rate for WD was significantly larger than that for montmorillonite. There was not enough evidence to conclude that the mean log K_ds among all cells of the two-way combinations of SS and presence or absence of AHA were the same (p-value = 0.6123); the log K_ds were different for SS in the presence and absence of AHA (experiment specific).

Sampling the exposures showed the flux of dissolved Ni versus sorbed Ni; Ni transposed compartments (sediment to water). The fifth and final set of analyses used Friedman's test [163] and multiple linear regressions to quantify the flux of Ni between compartments (dissolved and sorbed) and determine how K_d and survival are related to flux. In contrast to the amount of sorbed Ni at 48 h, the amount of dissolved Ni at 48 h was different among the four arenas (SS + Ni batch, SS + Ni SRF chamber, SS + Ni SRF channel, and SS + Ni + AHA batch) and three SS (WD, montmorillonite, and kaolinite). Neither the mean ranks of dissolved Ni among all cells of the two-way combinations of SS and arena (p-value = 0.0003), nor the mean ranks of dissolved Ni among the four arenas (p-value = 0.0053) were the same.

In fact, the mean ranks for both SRF arenas were significantly larger than the mean ranks for both batch arenas. Although the means could not be formally tested, this suggested that the means for the SRF arenas (chamber 574.3 and channel 582.0) were larger than the means for the batch arenas (379.7 for SS + Ni and 117.2 for SS + Ni + AHA). The mean ranks of dissolved Ni among the three types of SS were not the same (p-value = 0.0001). The mean rank for montmorillonite was larger than the mean ranks for both WD and kaolinite. Although the means could not be formally tested, it suggests that the mean for montmorillonite (1047.5) was larger than the means for WD and kaolinite (118.0 and 74.4, respectively) (Appendix F, Table 10). Mean ranks shaded by different colors are significantly different from each other. Using a level of significance $\alpha = 0.05$, there was not enough evidence to suggest that the mean ranks of sorbed Ni among all cells of the two-way combinations of SS and arena were different (p-value = 0.0729) (Appendix F, Table 11). No further tests were required.

As regards the relationship between log K_d and the 48 h measurements of dissolved and sorbed Ni, the predictors (dissolved and sorbed Ni measured at 48 h) account for 81 % (R-square = 0.8139) of the variability in the outcome log K_d; the model was significant (p-value = 0.0005). The remaining 19 % variability was attributed to one or more mitigating factors. Specifically, dissolved Ni at 48 h was a significant predictor (p-value = 0.0080). The parameter estimate equaled -0.0014, indicating that for each 100-unit increase in the amount of dissolved Ni at 48 h, the value of log K_d was expected to decrease by 0.14. Sorbed Ni at 48 h was also a significant predictor (p-value = 0.0035). The parameter estimate equaled 0.00087, indicating that for each 100-unit increase in the amount of sorbed Ni at 48 h, the value of log K_d was expected to increase by 0.087.

With respect to the relationship between the percent survival of *D. magna* and the amount of dissolved (time zero minus time 48) and sorbed (time 48 minus time zero) Ni measured at 48 h, the predictors (change in the amount of dissolved and sorbed Ni between the beginning and end of the experiments) accounted for 69 % (R-square = 0.6940) of the variability in the outcome percent survival; the model was significant (p-value < 0.0001). However, the change in the amount of dissolved Ni in

48 h was a significant predictor (p-value < 0.0001), while the change in the amount of sorbed Ni in 48 h was not a significant predictor (p-value = 0.0658). The parameter estimate for dissolved Ni in 48 h equaled 0.0391, indicating that for each 100-unit increase in the change in the amount of dissolved Ni in 48 hours, the value of percent survival was expected to decrease by 3.91 % (Appendix F, Figure 8).

DISCUSSION

Evaluating toxicity and quantifying sorption, research explored the independent and interactive effects of two aquatic stressors (Ni and SS). The primary research objective was to understand the flux of stressors (SS and Ni) between three compartments—water, sediment, and biota [164]. The flux of Ni as interpreted by the partition coefficient and the toxic effects of artificially contaminated materials to particle-feeding water fleas (*D. magna*, a biomonitor that integrated the stressors) were investigated by varying SS levels, sediment/clay type, Ni concentration, DOC content, and treatment scale. To investigate SS, contaminants, and HA in aquatic environments, clayey sediments, Ni, and AHA, respectively, were manipulated as fundamental stressors in various combinations in batch and SRF experiments. Artificially contaminated clay minerals (montmorillonite and kaolinite) and natural clayey sediment acted as adsorbents, sorbing Ni from aqueous solutions. Results illustrated that (suspended) solids and metals (e.g., Ni) act as stressors in aquatic systems and should be considered together as separate components and key factors in risk assessments. Furthermore, the addition of AHA diminished the toxicity of Ni.

Valid risk evaluations require the incorporation of SS and bioavailability into an assessment tailored to the hydrogeochemistry and biogeochemistry of the locale. The author agrees with NiPERA and the Danish Rapporteur (regulatory body assessing the risk of Ni for European member states) that an “eco-region” approach to regulation is appropriate [165]. Bioavailability and SS are significant because different waters and soils have different tolerances for Ni based on their chemistry. For instance, clay absorbs Ni much more readily than sandy soil, making it less accessible to plants and other organisms. Using an “eco-region” approach to regulation, data from systems typical of surface waters and sediments in Europe could be used to provide a range of safe Ni concentrations for other systems with similar chemical compositions [165]. The adoption of an ecosystem approach to the assessment of the aquatic environment necessitates integrated planning and monitoring of physical, chemical, and biological components [38].

These inquiries attempted to separate natural and anthropogenic stressors and predict their importance. Investigations of Ni-spiked clay minerals (montmorillonite and kaolinite) and Ni-spiked natural clayey sediment were performed in two different arenas. Positively charged molecules such as Ni may become substantial and detrimental components of the solid fraction suspended and resuspended in the water column, being bioavailable to aquatic organisms [76]. Hence, determination of the bioavailable fraction and the route of exposure are critical for a sound risk assessment [166].

A major ecosystem stressor, SS have been identified as the leading cause of impairment of the Nation’s waters [11] and also are important sites for contaminant assimilation and release [8],[9]. Interactions between solid and liquid phases in soils and aquatic systems result in sorption and desorption and consequent accumulation and mobility of environmental pollutants [65]. The magnitude of sorption depends on many factors, among them sediment type.

Select aquatic stressors for macroinvertebrates (specifically *D. magna*)

Sediment type

The type of SS influenced the degree of toxicity; the survival rate for *D. magna* in contaminated montmorillonite was significantly lower than in contaminated WD and kaolinite. Clay minerals are an important constituent of sediment since they play the role of natural scavenger by filtering out pollutants (through both ion exchange and adsorption mechanisms) from water [56]. Properties of clays that make then excellent materials for adsorption include the high specific surface area, chemical and mechanical stability, layered structure, high CEC, and Brönsted and Lewis acidity [102],[24],[68],[56],[110]. Even though WD was 75 % clay, autochthonous DOC and other characteristics for which WD was not analyzed lessened its apparent toxicity.

As expected, the adsorption capacity of montmorillonite generally was much higher than that of kaolinite. Bhattacharyya and Gupta [24],[93] observed that montmorillonite had a much higher initial rate of uptake than kaolinite, which might be due to very high specific surface area and CEC of montmorillonite as compared to kaolinite.

Additionally, montmorillonite has a net negative charge of 0.8 units per unit cell and this has been responsible for giving superior activity to montmorillonite as an adsorbent [24]. Active sites on the surface of clays (e.g., Brönsted and Lewis acid and base sites, ion exchange sites) lead them to be good adsorbents. As discussed by Puls and Bohn [68], the physical differences between kaolinite and montmorillonite may explain differences in sorption: (i) kaolinite has a greater percentage of hydroxyl edge sites, and (ii) montmorillonite has a larger percentage of ditrigonal cavities formed by six corner-sharing silica tetrahedral on the siloxane planar surface. Although kaolinite has a net zero layer charge, small negative charges at the broken edges are responsible for the activity.

In contrast to kaolinite, montmorillonite shows a greater distribution of charge and acts as a hard Lewis base. Relative localization of charge (strictly tetrahedral or octahedral substitution) tends to cause the formation of inner-sphere complexes, while greater distribution of charge (octahedral and tetrahedral substitution) tends to cause the formation of outer sphere complexes (with cations in solution) [167]. Kaolinite is a relatively soft Lewis base due to its hydroxyl edge surface functional groups [110]. For example, Abollino et al. [101] explained that one type of clay (e.g., kaolinite) may not adsorb metals as well as another type of clay (e.g., montmorillonite) because complex formation by ligands (EDTA and other acids) hindered the sorption of the metals.

Suspended solids

Suspended solids are a ubiquitous water pollutant, causing significant environmental damage and economic costs [35],[168],[29]. Suspended sediments have a multitude of potential environmental impacts on water bodies, including transport of other pollutants, notably sorbed trace elements and toxic organics [29],[35]. Many toxic substances entering aquatic ecosystems accumulate in the bottom sediment, which constitutes a large reservoir of potentially bioavailable contaminants [169]. Bottom sediments and associated contaminants can be resuspended into the water column by human activities such as and vessel passage [53],[168] or by natural processes such as flow, bioturbation, and wind-induced turbulence [170],[171],[172],[173].

Results of the SS only batch experiments agreed with the work of Weltens et al. [76]. The experiments showed that uncontaminated solids used at test concentrations up to 250 mg L^{-1} caused no significant mortality within the 48 h of exposure [76]. The experiments established that the three uncontaminated SS used at the maximum test concentration of 50 NTU caused no significant mortality during the 48 h exposure.

Nevertheless, the experiments involving spiked sediment confirmed that the exposure of aquatic macroinvertebrates to contaminated, suspended particles may produce significant acute effects. Routes of administration included dissolved and sorbed Ni. The general assumption is that the dissolved fraction of a toxic substance in surface water is mainly responsible for toxicity to aquatic organisms [76]. As turbidity increased, more dissolved Ni at time zero was present and bioavailable. However, Ni also was adsorbed to suspended particles in the water column. In response to different physicochemical conditions, the adsorbed metals may desorb in the gastrointestinal tract and exert toxic effects [76]. Robinson and Klaine (Sarah E. Robinson and Stephen J. Klaine, Clemson University, Clemson, SC, USA, personal communication) found that *D. magna* cleared its gut tract of kaolinite faster than for montmorillonite (approximately 30 minutes versus 60 minutes, respectively).

Results demonstrated that suspended contaminated particles exert a toxic effect that cannot be explained by their physical presence or the dissolved concentration of Ni. For instance, 23 and 33 % survival, respectively, was seen with contaminated montmorillonite in 50 NTU batch SS + Ni and in 50 NTU SRF chamber/channel SS + Ni, although the dissolved concentration was not toxic in water-only exposure (1266 and 1511/1495 µg L^{-1}, respectively). Thus, the type of SS and the sorbed Ni cause mortality. Evidently, the particle-bond fraction of the contaminant became available within the body.

The results of this research suggest both SS and HA are important in a Ni BLM [136],[134]. Specifically, the present research contributes evidence to support the Ni BLM and to incorporate SS into the models. The BLM incorporates aqueous speciation reactions and competition of cations for binding to biotic receptors [86]. All aspects of water chemistry that affect toxicity can be included in the BLM since it integrates the concept of bioavailability into site-specific ambient water quality criteria [133]. This research focused on investigating the effects of various water parameters (type of sediment, SS, and DOC), which could be useful in refining the present models. As demonstrated in batch experiments, DOC was a critical aquatic parameter that alleviated Ni toxicity.

Ni toxicity and attenuation of toxicity by DOC (as AHA)

DOM has a strong affinity for Ni and is known to reduce the bioavailability of metals in aquatic systems [79]. Dissolved HSs are dynamic materials [65]. Their chemical and structural characteristics depend on the aqueous chemistry of the system. Glover et al. [120] found that aromatic carbon content may govern the ameliorative actions of NOM. Humic substances on the one hand enhance the solubility of sparingly soluble substances but, on the other hand, reduce their bioavailability probably by complexation [65],[174]. Divalent cationic trace metal ions, such as Ni(II), are known to complex with DOM (e.g., [73],[74],[116],[175]). In situations where the free metal ion is the main bioavailable species, complexation with DOM therefore will alter metal bioavailability and toxicity to aquatic organisms (see review by [63]).

Generally among all experiments, rates of survival of *D. magna* exposed to WD were higher than those of montmorillonite and kaolinite, perhaps indicating that the NOM in WD attenuated the toxicity of Ni. Furthermore, DOC in the form of AHA was expected to attenuate, and in fact did, the effects of Ni toxicity (e.g., SS + Ni + AHA, Ni + AHA).

When AHA was added to turbid suspensions of contaminated WD and montmorillonite, HA immobilized the Ni(II), increasing the adsorption capacity of the SS and reducing the desorption of Ni.

Organic carbon present in WD sediment and in the form of added AHA mitigated the toxicity of Ni to *D. magna*, corroborating the research of Arias et al. [176], among others. The ability of HSs to bind, and therefore detoxify, environmental metal pollutants is well described (e.g., [134]). Research by Glover et al. [120],[121] showed that HSs actually have highly variable protective effects on silver toxicity to *D. magna*, probably due to different complexation affinities for the metal toxicant.

But the results of experiments involving AHA were not always as expected. Martino et al. [78] noted the competing effects of DOM and particle sorption sites for dissolved Ni. Arias et al. [176] showed that as HA concentration in the sorbent increases, the sorption of Hg(II) also increases, except for low Hg(II) concentrations and high concentrations of DOM. Working with kaolin, Arias et al. [176] also theorized that the DOM released from the solid fraction to the soil solution may act as a competitive ligand, possibly increasing the mobility of Hg. This may explain why even higher survival was not the case in experiments involving the addition of 10 mg L^{-1} AHA to WD and montmorillonite.

In the Ni + AHA batch experiments, increasing concentrations of AHA resulted in increased survival, despite the toxic Ni concentration (LC_{75}), yet only up to a point. Higher rates of survival would be expected since the presence of more DOC theoretically would attenuate the toxicity of Ni to *D. magna*. Nevertheless, the author suspects that as the concentration of HA increased, the *D. magna* were inhibited from moving/feeding by the sticky HA clinging to their appendages. A physical preponderance of HA debris caused the demise of the test organism. The synergism of the Ni LC_{75} and the relatively 'high' AHA concentration caused the survival of *D. magna* to decrease rather than increase at higher levels of HA. Indeed, the AHA only batch experiments indicated that higher levels of AHA actually decreased survival of *D. magna*. Results of the characterization of colloidal humic-bound Ni and uranium in the "dissolved" fraction of contaminated sediment extracts [177] suggested that Ni was potentially bioavailable because the humic-bound Ni was mainly present as labile complexes or the free cation.

While DOM can sequester metal ions in solution, it can also facilitate the release of metal ions adsorbed to sediments [65]. A good positive correlation often has been observed between the contents of organic materials and metal concentrations in aquatic sediments [65]. Moreover, differences in Ni binding may occur due to the DOM source, the content of the HS, and/or the FA/HA ratios within a given DOM sample [42]. The HA fraction complexed Ni to a greater extent than did the FA fraction [42]. The concentration of DOC played a more significant role than either DOC source or fraction in determining Ni speciation and therefore bioavailability and toxicity to *H. azteca* [79]. Doig and Liber [42] concluded that the "degree of influence on speciation was determined primarily by the Ni:DOC ratio rather than the environmental source of DOM."

The similarities and differences among the results of the various experiments (Ni only, Ni + AHA, SS only, SS + Ni, and SS + Ni + AHA) can be attributed to one or more mitigating factors, such as the sorption capacity of sediment, the presence of organic carbon, pH, or water hardness. This research confirmed that metal molecules, attached to solid material and dissolved in the water column, are a source of metal toxicity to filter feeding organisms. The higher toxicity results for montmorillonite as compared to WD and kaolinite strongly suggest that the fraction adsorbed on this clay becomes available to the filter-feeding test organism, perhaps through desorption in the gut [143].

Possible reasons for scale differences between batch and SRF

Microcosms such as the SRF can provide realistic stressor exposures under environmental conditions that are difficult to reproduce in the laboratory [178]. Artificial streams provide control over relevant environmental variables and allow for the separation of multiple stressors contained within complex effluents [179],[180],[181],[182],[183],[184]. The flume can be considered a good surrogate for a straight reach of stream.

In the SRF experimental treatment, we tested the hypothesis that the cladoceran *D. magna* would experience less toxicity than in the batch experiments from exposure to the same level (50 NTU) of Ni spiked sediment/clay (WD, montmorillonite, and kaolinite). SRF experiments also attempted to discern how Ni fluxed from spiked slurry and whether this flume treatment differed from batch results. Since variations in physicochemical parameters such as pH, water hardness, alkalinity, and temperature have all been shown to increase or decrease sensitivity to toxicants [181],[86], these differences were minimized among all experiments in both arenas (batch and SRF).

Indeed, survival depended on exposure conditions, with higher survival in the SRF versus batch possibly due to advective flow (albeit recirculating). Some other possible reasons for differences between arenas are the availability of refugia in the SRF, differences in the dissolved versus the sorbed fraction (dissolved is theoretically more bioavailable), and the increased size and heterogeneity of the SRF system. Results for SRF channel arena were more consistent with the batch experimental arena than with the SRF chamber arena. In addition, the partition coefficients for the three arenas were unique to each experiment.

Flux of dissolved and sorbed Ni

Speciation and flux are dynamic phenomena; dynamics is by definition an integration of space and time. The sorption coefficient is strictly an equilibrium concept [67]. Determining the K_d helps illuminate the solid-liquid distribution and speciation of Ni in the suspension. In both batch and SRF experiments, a valid assumption is that within 48 h local equilibrium between the metal solutes and sorbents was attained [67]. The calculated log K_ds generally agreed with the values compiled and calculated by Allison and Allison [2].

The flux of Ni between compartments was evident in all experiments; Ni transposed compartments. Water column concentrations reflected Ni desorption from the spiked sediment. Ni desorbed from the spiked sediment/clay rather quickly, within minutes as gleaned from dissolved concentrations at time zero. Even so, Ni adsorbed back onto the clay particles. Since stability is associated with an ordered arrangement, Ni(II) ions in aqueous solution are in much more chaotic distribution than they are in the adsorbed state. Ni(II) thus will have strong affinity towards clays [91]. Clay minerals were capable of scavenging Ni from aqueous media by behaving as adsorbents. Comparable to Gupta and Bhattacharyya [93], these experiments support the use of montmorillonite and kaolinite for removing Ni from aqueous media through adsorption-mediated immobilization. Identical to their findings, montmorillonite sorbed more Ni than kaolinite [56],[92],[24].

No discernable trends were found for where (i.e., compartment) Ni resided after equilibrium, though more Ni seemed to be dissolved rather than sorbed. The amount of dissolved Ni (but not sorbed Ni) was different among the four arenas and three sediment/clays, suggesting that the mean concentrations for the SRF arenas and for montmorillonite were larger than the others. The empirical model used to predict K_d revealed that partitioning of Ni between the solid and solution phases was experiment specific. Ions such as Cl^- and SO_4^{2-} competed with the mineral surfaces for the divalent metal cations [68],[105],[107].

Not surprisingly, the predictors of log K_d (dissolved and sorbed Ni at time 48 h) accounted for 81 % of the variability of the outcome of this parameter. Similarly, the predictors (change in the amount of dissolved and sorbed Ni between the beginning and end of the experiments) accounted for 69 % of the variability of the outcome of percent survival. Even so, for mean percent survival, the change in the amount of dissolved Ni (significant) had more influence than the change in the amount of sorbed Ni (not significant). Other factors in this complex system account for the remainder of the unpredictability in the outcomes.

Caveats of this research

Perhaps the most pressing proposal for future research is to address the caveats of this research. Physical, geochemical, and biological processes all contribute to the differences inherent in nature. One component of the ecosystem, sediment, composed of mineral and organic materials, often exhibits high spatial and temporal variability [36],[37]. This research attempted to isolate stressors in a simplified scenario, in contrast to the complexity and changeability in the environment [3],[33],[37]. Using batch and SRF experimental treatments helped control for the environmental variability that affects exposure of organisms and ecological systems to stressors [37]. Consequently, spatial and temporal scaling issues were minimized. However, although the relatively controlled conditions of laboratory experiments are necessary to separate stressors, they are not particularly reflective of nature. Indeed, the route of administration in ecotoxicity tests is paramount. To correctly evaluate the potential risk for filter feeders, a flow-through system is needed to ascertain the effect of adsorbed molecules [76].

Even though laboratory streams can be utilized to investigate a wide array of environmental perturbations, the reliability of laboratory streams to predict effects in nature is “questionable” [183],[182]. The recirculating water of the SRF makes that microcosm more representative of a lentic ecosystem than a lotic system [183]. Artificial stream research cannot be generalized to all streams because toxicants behave differently in water with different characteristics. Compromises must be made between complex systems and more simple systems with “high degrees of reproducibility and control” [183]. In particular, researchers utilizing artificial streams should consider the effects of bias errors in experimental design, data acquisition, data reduction, and data interpretation [185]. Kosinski [182] proposed that the two best steps to strengthen future toxicological artificial-stream research are the replication of streams within treatments and more attempts to perform an integrated, comparative series of single-species, artificial-stream, and field experiments.

Furthermore, dynamism is more prevalent than true equilibrium in nature. Therefore, equilibrium-based models and equilibrium-based sediment quality guidelines are not always realistic. In nature, rapid physicochemical and microbiological alterations may occur in the sediment that might alter bioavailability and toxicity [14]. Unfortunately, no risk assessment studies all the fate processes between waters and sediments (e.g., resuspension/deposition, advection, upwelling/downwelling, diffusion, bioturbation, diagenesis, and bioaccumulation) [166]. Valuable insight would be gained from examining further not only SS, but also bedded sediment and the hyporheic zone (sediment-water interface), utilizing a variety of test and wild-caught organisms. More research is needed to establish the relative importance of pore water and overlying water as exposure routes for Ni to aquatic biota.

Not all bioavailability factors (e.g., AVS, SEM-Ni, Fe, Mn, and redox) were monitored in this research. Nonetheless, in a natural system in non-equilibrium, the free Ni ion may still best predict bioavailability because Ni has exhibited slow ligand exchange kinetics [42],[64]. To address these dynamic processes and place this research in proper context, a Weight-of-Evidence (WOE) approach [186],[13],[187],[188] that integrates multiple lines-of-evidence (LOE) to establish cause/effect relationships for multi-stressor environments would be ideal.

Besides WD sediment, this study used relatively pure clay minerals rather than "whole" natural media for experimentation and the empirical determination of partition coefficients. By and large, clays typically do not exist in such pure forms in nature. Metals and organic ligands combined with clays react in a variety of ways depending on the physical, chemical, and biological conditions and the accompanying physicochemical parameters [101],[100],[80],[189],[71]. The results seen with the analytical clays used in this research may not occur in the same way or as markedly in nature. Testing additional types of “whole” natural media and low-cost sorbents and using low metal concentrations where K_d is less likely to depend on metal concentration would address conditions more representative of nature.

From the perspective of a chemist or risk assessor, many of the particle-phase concentrations measured from the experiments are unreasonably high compared with those existing in the environment. Therefore, partitioning in these tests may not be representative of Ni K_ds in systems where levels of total Ni are less. The experimental conditions thus likely represented a worst-case scenario of environmental Ni exposure.

Nor was the work verified in the field/*in situ*. *In situ* tests are more realistic than laboratory tests because they reduce the artifacts associated with grab sampling and lab manipulations, and because they are better at capturing fluctuating exposures and stressor interactions [52],[190],[32]. In contrast to the batch experimental treatment, the SRF better simulated natural conditions, but still was far inferior to *in situ* assays. An idea for future study is to expand the research to the mesocosm and field (*in situ* exposures) using an integrated, comparative approach [182],[52],[190],[40],[32].

Solids and flow are intertwined [191],[192]. Although SS and flow are so closely interrelated that under natural conditions it frequently is impossible to separate the individual effects of the two [193],[5], flow was not manipulated or studied in this research. The approach used to monitor the SS (i.e., turbidity) also is suspect. Henley et al. [34] caution, "The use of NTU as a surrogate measurement of suspended sediment to predict biotic effects within watersheds is dubious" because of the lack of correlation between suspended sediment concentrations (mg L^{-1}) and units of measure (NTU) among watersheds and experiments.

The mixture of AHA, M^{n+}, and reconstituted water (containing Ca, Mg, etc.) was not "aged". At the beginning of each test, organisms probably were exposed to higher M^{n+} concentrations than those that would exist under some real-world circumstances [86]. Aging the mixture would allow the metal in the 'receiving water' to displace Ca and Mg and equilibrate with the DOM, making the mixture less toxic [86]. Likewise, the results of Glover et al. [120] suggest that the degree of amelioration is dependent on source of NOM and on equilibration time. An increased equilibration time resulted in decreased toxicity and decreased whole-body silver accumulation [120], a finding that may be applicable to Ni. Since Ni has a relatively high stability constant, equilibration time for Ni is very important. Ni equilibrated slowly with natural DOM in hard freshwater [64]. According to Xue et al. [64], Ni speciation may never reach equilibrium in natural waters because of slow ligand-exchange kinetics. The higher the DOC level in water, the more ligands present to bind metals.

Differences in Ni binding may occur due to DOM source and composition, as occurred when DOC source influenced binding between metals and OC [42]. In surface waters, FAs constitute 80 % of the DOC, while HAs constitute 20 % of the DOC of HSs [127]. Glover and Wood [61] tested various types of NOM, including AHA, and discovered that commercially available HSs in laboratory tests may not be ecologically applicable [120]. These findings have significant implications for the study of environmental metal toxicology [61]. Actually, Glover et al. [120],[61] caution that the actions of HSs on daphnid sodium metabolism also are source dependent. Inhibition of sodium metabolism may be the major mode of toxic action [119],[61],[194],[195],[196]. "Actions of humic substances on sodium metabolism could exacerbate or counter toxic effects in competition or collaboration with the beneficial effects of metal ion chelation by humic matter" [61].

Furthermore, Doig and Liber [42] found differences in Ni speciation resulted from variable HS content or different FA/HA ratios within a given DOM sample. The HA fraction complexed more Ni, and more strongly, than the corresponding FA fraction at a give Ni:DOC ratio [42]. Even so, on the whole, the concentration of DOC exerted greater control than either DOC fraction or source in determining Ni speciation and thus bioavailability and toxicity [79]. Studying Ni sequestration in a clay mineral-humic acid complex (as did Nachegaal and Sparks [197] with kaolinite-humic acid complexes, or as did Arias et al. [176] with mercury and kaolinite-humic acid complexes) would advance the purpose of remediation.

This research did not examine the effect of pH on adsorption-desorption, as did Arias et al. [176]. The role of HA in the immobilization of Hg(II) is fundamental, increasing the adsorption capacity of kaolin. This effect is especially important for an acid medium because the Hg(II) desorption for kaolin at $pH = 2.5$ is > 50 %. The presence of HA dramatically reduces these desorption percentages to values < 1 %, and hence the quantity of Hg(II) that remains in water solution, preventing toxicity problems in surface and subsurface waters [176]. The adsorption isotherms showed that as HA concentration in the sorbent increases, the sorption of Hg(II) also increases, except for low Hg(II) concentrations and high concentrations of DOC [176]. This reflects the importance of DOM in the Hg(II) sorption processes, and how the DOM released from the solid fraction to the soil solution may act as a competitive ligand, possibly increasing the mobility of Hg [176].

Many sources of uncertainty exist for metal partition coefficients, principally because they are site-specific. For instance, partition coefficients vary with pH and with the concentration of sorbing phases in the soil matrix. Although DOC/TOC was measured for the relevant experiments, the weight percent of hydrous ferric oxides and corresponding oxides of aluminum and manganese for WD sediment were not determined. Dissolved ligands can and did complex with metals, reducing their propensity for sorption in proportion to the concentration of the ligands. When more than one metal is present, metals compete for sorption sites, as ligands do for complexation.

Other sources of uncertainty include limits in the maximum K_d caused by metal concentration detection limits, non-attainment of equilibrium, variability in the methods of measurement, variability in the extractants used in batch tests, redox conditions, and neglect of the impact of total system metal concentration on the magnitude of K_d [2]. Since K_d is site/experiment-specific and dynamic, investigating metal partitioning in runoff water, in the suspended sediment load of lentic and lotic systems, between riverine or lacustrine sediment and its pore water, and between DOC and inorganic solution species [2] would be informative.

CONCLUSIONS

Results support the hypothesis that SS and Ni act as stressors in streams. The hydrogeochemistry/biogeochemistry of the milieu clearly affects the flux of Ni. Beyond its direct connection with bioavailability, Ni fate is dominated by sediment type, solids level, distribution, and speciation. Both the type of sediments and the concentration of SS affect the toxicity of exchangeable Ni. Suspended solids should be considered as a separate compartment and key factor in risk assessments.

Monitoring of the turbidity levels and the total, dissolved, and sorbed concentrations of pollutant metals is a constructive approach for the integration of chemical partitioning and sediment dynamics. The addition of AHA to this flux further portrays the variability inherent in nature. Contaminated mineral materials as well as organic materials contributed to particle-bound and water column toxicity. Beyond aqueous concentrations, metal toxicity is driven by a complex mixture of metal bound ligands. The toxicity of metals to aquatic organisms decreases when cationic metals form complexes with organic matter and inorganic anions [134],[136],[137],[133].

Though difficult to remove, persistent pollutant metals can be removed from water through adsorption by a variety of materials, especially clays [91],[25],[56],[55], Gupta et al. in press, [59],[95]. "Adsorption on a suitable material has been a preferred method with high potential for removal, recovery and recycling of metals from wastewater" [91]. The chief concerns in the development of these materials are low capital cost, suitability for both batch and continuous processes, ease of operation, little or no secondary pollutant generation, applicability at very low metal concentrations, improved treatment efficacy, and possibility of regeneration and reuse [91],[198].

Clay minerals were capable of removing Ni from aqueous media by behaving as metal sorbents. This study suggests montmorillonite as compared to kaolinite would be more effective for adsorbing Ni discharges. Even though montmorillonite and kaolinite differ in their ability to sorb Ni, both montmorillonite and kaolinite have good potentialities as low-cost natural sorbents, as Abollino et al. [100] found for montmorillonite and vermiculite.

Sampling the exposure showed the flux of dissolved Ni versus sorbed Ni. Easily desorbed Ni was released rather quickly from the SS, as shown in water column Ni concentrations. Ni transposed compartments (sediment to water); the water column concentration reflected Ni desorption from spiked sediment. Higher concentrations of dissolved Ni were present in the two SRF arenas (chamber and channel) and for montmorillonite. While the change in the amount of sorbed Ni was not a significant predictor, the change in the amount of dissolved Ni was a significant predictor of percent survival. This suggests that the concentration of dissolved Ni is a good indicator of bioavailability and hence toxicity. Yet, ultimate toxicity depended on the type of suspended solid, with highest mortality imparted by montmorillonite.

Moreover, the calculation of the solid-liquid distribution coefficient (K_d) (between dissolved Ni and sorbed Ni at 48 h) revealed a substantial difference between WD/montmorillonite and kaolinite, which may be attributed to properties of the different clays. Additionally, a significant difference for K_d and dissolved Ni was discerned for batch/SRF channel as compared to SRF chamber. Overall, partition coefficients were experiment specific, making K_ds difficult to predict and evaluate.

Concisely, Ni toxicity to *D. magna* was mitigated by OC, advective flow, and scale. For those systems with theoretically available Ni, the lack of mortality was attributed to one or more mitigating factors, including the presence of organic carbon, pH, or water hardness. Autochthonous HA and AHA attenuated Ni toxicity, probably through complexation. Undeniably, the DOM in WD sediment had an attenuating effect on Ni toxicity; this influence was evident for WD as compared to montmorillonite and kaolinite. Likewise, the addition of AHA to suspensions and solutions attenuated the toxicity of Ni. Nonetheless, percent survival of the test organism was reduced at higher AHA concentrations in the AHA only tests. As Martino et al. [78] note, "Given the profound implications for biogeochemical modeling…the effects merit further investigation for a broad suite of metals adsorbed under a wide variety of natural conditions".

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APPENDICES

Appendix A: Research summary

Appendix table 1. Comprehensive summary of experimental methods, treatments, and measurements

Appendix table 1. Comprehensive summary of experimental methods, treatments, and measurements

METHODS				TREATMENTS							
Arena	Duration	EXPERIMENT	TEST NUMBER	Nominal [Ni]	Actual [Ni]	Aldrich Humic Acid (AHA)	SS Type	Turbidity/Total Suspended Solids Concentration			SURVIVAL
				mg kg^{-1} or µg L^{-1}	mg kg^{-1} or µg L^{-1}	mg L^{-1}		as Nominal NTU	as Actual NTU	as mg L^{-1}	% ± 1 SD
Batch/4 L beakers	48 h	Ni only	1	200	207	n/a	n/a	n/a	n/a	n/a	100 ± 0
			2	375	345						83 ± 5
			3	750	750						65 ± 6
			4	1500	1516						53 ± 5
			5	3000	3079						15 ± 6
			6	6000	6153						0
		SS only	7	n/a	n/a	n/a	WD	50	49.8	66.00	100 ± 0
			8				Mont	50	49.7	248.63	100 ± 0
			9				Kaol	50	50.1	51.23	100 ± 0
		SS + Ni	10	5000	5162	n/a	WD	12.5	12.4	14.23	100 ± 0
			11					25	25.0	36.63	90 ± 8
			12					50	50.3	66.00	75 ± 6
			13	5000	4094	n/a	Mont	12.5	12.6	72.86	43 ± 10
			14					25	25.2	123.70	33 ± 10
			15					50	50.4	248.63	23 ± 10
			16	5000	3599	n/a	Kaol	12.5	12.7	7.90	93 ± 5
			17					25	24.9	18.63	80 ± 8
			18					50	49.8	51.23	65 ± 10
SRF/480 L (127 gal)	48 h	SS + Ni in chamber	32	5000	5162	n/a	WD	50	32.1	66.00	85 ± 6
			33		4094		Mont	50	23.0	248.63	33 ± 10
			34		3599		Kaol	50	38.4	51.23	73 ± 10
		SS + Ni in channel	32	5000	5162	n/a	WD	50	32.1	66.00	85 ± 6
			33		4094		Mont	50	23.0	248.63	33 ± 10
			34		3599		Kaol	50	38.4	51.23	73 ± 10
Batch/4 L beakers	48 h	SS + Ni + AHA	19	5000	5162	10	WD	50	53.5	66.00	93 ± 10
			20		4094		Mont	50	53.9	248.63	50 ± 8
		Ni + AHA	21	2150 (LC_{75})	2201	0	n/a	n/a	0.1	n/a	25 ± 6
			22		2188	1			0.7		25 ± 6
			23		2196	10			3.0		28 ± 10
			24		2191	25			6.7		43 ± 5
			25		2112	60			14.0		30 ± 8
			26		2097	100			20.0		15 ± 6
		AHA only	27	n/a	n/a	1	n/a	n/a	0.7	n/a	100 ± 0
			28			10			3.1		100 ± 0
			29			25			6.6		95 ± 6
			30			60			13.9		85 ± 6
			31			100			19.9		45 ± 6

RESULTS										
TOTAL Ni (µg L^{-1})		**DISSOLVED Ni (µg L^{-1})**		**SORBED Ni (µg L^{-1})**		**Mean DOC (mg L^{-1})**		**TOC**	**Kd [Ni]**	**log Kd [Ni]**
Time 0 h	**Time 48 h**	**Time 0 h**	**Time 48 h**	**Time 0 h**	**Time 48 h**	**chamber**	**channel**	**%**	**L kg^{-1}**	**(L kg^{-1})**
207	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
345										
750										
1516										
3079										
6153										
n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	7.66	n/a	n/a
								0.05		
								0.05		
83	70	51	66	32	4	0.20	n/a	7.66	4259.03	3.63
220	212	162	150	58	62	0.36			11284.01	4.05
517	399	332	289	185	110	0.41			5767.01	3.76
146	130	142	128	4	2	0.00	n/a	0.05	214.45	2.33
552	450	508	433	44	17	0.03			317.39	2.50
1369	603	1266	192	103	411	0.09			8609.68	3.93
56	55	57	55	0	0	0.04	n/a	0.05	n/a	n/a
109	105	108	102	1	3	0.07			1578.73	3.20
219	199	213	191	6	8	0.09			817.58	2.91
716	2037	420	241	296	1796	0.46	n/a	7.66	112913.37	5.05
1696	1328	1511	73	185	1255	0.00		0.05	69146.04	4.84
480	783	425	319	55	464	0.04		0.05	28392.45	4.45
657	398	430	240	227	158	n/a	0.71	7.66	9974.75	4.00
1642	465	1495	62	147	403		0.00	0.05	26143.27	4.42
493	367	438	315	55	52		0.04	0.05	3222.32	3.51
654	531	457	397	197	134	7.88	n/a	7.66	5114.11	3.71
1463	1197	1287	1042	176	155	7.00		0.05	598.29	2.78
2201	n/a	n/a	n/a	n/a	n/a	0.42	n/a	n/a	n/a	n/a
2188						1.48				
2196						7.14				
2191						18.27				
2112						48.02				
2097						80.01				
n/a	n/a	n/a	n/a	n/a	n/a	1.36	n/a	n/a	n/a	n/a
						7.38				
						17.22				
						48.77				
						79.03				

Appendix B: Data for the calculation of partition coefficients

Appendix table 2. Batch and SRF summary table for turbidity, TSS, and distribution coefficients for three clayey sediments

Appendix table 2. Batch and SRF summary table for turbidity, TSS, and distribution coefficients for three clayey sediments

BATCH: TURBIDITY, TSS & DISTRIBUTION COEFFICIENTS (K_d) FOR THREE CLAYEY SEDIMENTS									
Sediment Type	**Turbidity (NTU)**	**Mean TSS (mg L^{-1})**	**Mean TSS (kg L^{-1})**	**Reciprocal TSS (L kg^{-1})**	**Sediment-Ni (µg L^{-1})**	**Sediment-Ni (µg kg^{-1})**	**Water-Ni (µg L^{-1})**	**K_d, Distribution Coefficient (L kg^{-1})**	**log K_d**
WARDEN DITCH	12.5	14.23	0.00001423	70257.78	4	281031.1	66	4258.05	3.63
	25	36.63	0.00003663	27297.57	62	1692449.22	150	11282.99	4.05
	50	66	0.000066	15151.52	110	1666666.67	289	5767.01	3.76
MONTMORILLONITE	12.5	72.87	0.00007287	13723.69	2	27447.38	128	214.43	2.33
	25	123.7	0.0001237	8084.07	17	137429.26	433	317.39	2.5
	50	248.63	0.00024863	4021.99	411	1653036.82	192	8609.57	3.93
KAOLINITE	12.5	7.9	0.0000079	126582.28	0	0	55	n/a	n/a
	25	18.63	0.00001863	53667.36	3	161002.08	102	1578.45	3.2
	50	51.23	0.00005123	19518.56	8	156148.44	191	817.53	2.91
SRF CHAMBER: TURBIDITY, TSS & DISTRIBUTION COEFFICIENTS (K_d) FOR THREE CLAYEY SEDIMENTS									
Sediment Type	**Turbidity (NTU)**	**Mean TSS (mg L^{-1})**	**Mean TSS (kg L^{-1})**	**Reciprocal TSS (L kg^{-1})**	**Sediment-Ni (µg L^{-1})**	**Sediment-Ni (µg kg^{-1})**	**Water-Ni (µg L^{-1})**	**K_d, Distribution Coefficient (L kg^{-1})**	**log K_d**
WARDEN DITCH	50	66	0.000066	15151.52	1796	27212121.21	241	112913.37	5.05
MONTMORILLONITE	50	248.63	0.00024863	4022.04	1255	5047661.18	73	69146.04	4.84
KAOLINITE	50	51.23	0.00005123	19519.81	464	9057193.05	319	28392.45	4.45
SRF CHANNEL: TURBIDITY, TSS & DISTRIBUTION COEFFICIENTS (K_d) FOR THREE CLAYEY SEDIMENTS									
Sediment Type	**Turbidity (NTU)**	**Mean TSS (mg L^{-1})**	**Mean TSS (kg L^{-1})**	**Reciprocal TSS (L kg^{-1})**	**Sediment-Ni (µg L^{-1})**	**Sediment-Ni (µg kg^{-1})**	**Water-Ni (µg L^{-1})**	**K_d, Distribution Coefficient (L kg^{-1})**	**log K_d**
WARDEN DITCH	50	66	0.000066	15151.52	158	2393939.39	240	9974.75	4
MONTMORILLONITE	50	248.63	0.00024863	4022.04	403	1620882.44	62	26143.27	4.42
KAOLINITE	50	51.23	0.00005123	19519.81	52	1015030.26	315	3222.32	3.51

Appendix C: Data for the conversion of turbidity to TSS

Appendix table 3. Measurements for the conversion of turbidity (NTU) to TSS (mg L^{-1})

Appendix table 3. Measurements for the conversion of turbidity (NTU) to TSS (mg L^{-1})

DETERMINING CONVERSION OF TURBIDITY (NTU) TO TSS (mg L^{-1})													
Sediment/clay type	**Turbidity (NTU)**	**Replicate**	**Lab ID**	**Filter & pan weight (g)**	**Wet sediment/clay on filter in pan (g)**	**Sediment/clay wet weight (g)**	**Mean wet weight (g)**	**Mean wet weight (mg)**	**Dry sediment/clay on filter in pan (g)**	**Sediment/clay dry weight (g)**	**Mean dry weight (g)**	**Mean dry weight (mg)**	**Difference wet-dry (g)**
WARDEN DITCH	12.5	1	1	1.379	1.430	0.052	0.056	55.867	1.389	0.010	0.014	14.233	0.041
		2	2	1.366	1.424	0.058			1.382	0.016			0.042
		3	3	1.375	1.434	0.058			1.392	0.016			0.042
	25	1	4	1.338	1.464	0.126	0.122	122.000	1.380	0.042	0.037	36.633	0.084
		2	5	1.366	1.488	0.123			1.400	0.034			0.088
		3	6	1.353	1.470	0.117			1.387	0.034			0.084
	50	1	7	1.370	1.541	0.171	0.172	171.900	1.439	0.069	0.066	66.000	0.102
		2	8	1.373	1.529	0.156			1.435	0.062			0.094
		3	9	1.374	1.563	0.189			1.442	0.067			0.122
MONTMORILLONITE	12.5	1	10	1.353	1.520	0.167	0.168	167.667	1.425	0.072	0.073	72.867	0.095
		2	11	1.333	1.500	0.166			1.405	0.072			0.094
		3	12	1.362	1.531	0.170			1.436	0.075			0.095
	25	1	13	1.366	1.661	0.295	0.289	288.867	1.492	0.126	0.124	123.700	0.169
		2	14	1.335	1.625	0.290			1.459	0.125			0.166
		3	15	1.353	1.634	0.282			1.473	0.121			0.161
	50	1	16	1.314	1.889	0.575	0.586	585.867	1.551	0.237	0.249	248.633	0.338
		2	17	1.357	1.943	0.586			1.609	0.251			0.335
		3	18	1.373	1.970	0.597			1.630	0.257			0.339
KAOLINITE	12.5	1	19	1.350	1.379	0.030	0.029	29.433	1.358	0.009	0.008	7.900	0.021
		2	20	1.388	1.416	0.028			1.395	0.006			0.022
		3	21	1.381	1.411	0.030			1.390	0.009			0.022
	25	1	22	1.341	1.404	0.063	0.063	62.933	1.360	0.019	0.019	18.633	0.044
		2	23	1.360	1.423	0.063			1.379	0.019			0.044
		3	24	1.342	1.405	0.063			1.360	0.018			0.045
	50	1	24	1.349	1.520	0.171	0.177	176.467	1.392	0.043	0.051	51.233	0.128
		2	26	1.386	1.564	0.178			1.441	0.055			0.123
		3	27	1.333	1.514	0.181			1.389	0.056			0.125

Appendix D: Physicochemical parameters for batch and SRF experiments

Appendix table 4. Average DOC, pH, and temperature for selected batch and SRF experiments
Appendix table 5. Summary physicochemical parameters for 31 batch and 3 SRF experiments
Appendix table 6. Detailed physicochemical parameters for 31 batch experiments
Appendix table 7. Detailed physicochemical parameters for 3 SRF experiments

Appendix table 4. Average DOC, pH, and temperature for selected batch and SRF experiments

AVERAGE DOC, pH, AND TEMPERATURE FOR SELECTED BATCH & SRF EXPERIMENTS								
Sediment Type	**Turbidity (NTU)**	**Mean DOC (mg L^{-1})**			**Mean pH (units)**		**Mean Temperature (°C)**	
		Batch	**SRF Chamber**	**SRF Channel**	**Batch**	**SRF**	**Batch**	**SRF**
WARDEN DITCH	12.5	0.20	/	/	7.95	/	21.4	/
	25	0.36	/	/	7.84	/	21.8	/
	50	0.41	0.46	0.71	7.78	8.36	21.2	21.9
MONTMORILLONITE	12.5	0.00	/	/	8.01	/	22	/
	25	0.03	/	/	7.95	/	22.1	/
	50	0.09	0.00	0.00	7.98	8.00	20.9	22.3
KAOLINITE	12.5	0.04	/	/	7.75	/	21.3	/
	25	0.07	/	/	7.76	/	21.4	/
	50	0.09	0.04	0.04	7.83	8.33	21.8	22.4

Appendix table 5. Summary physicochemical parameters for 31 batch and 3 SRF experiments

Physicochemical parameters measured for 31 batch and 3 SRF experiments. Reported as the mean ± standard deviation (SD), minimum, and maximum values.

Test	Statistic	pH (units)	Temperature (°C)	Turbidity (NTU)[a]	DO[b] (mg L^{-1})	Specific conductance (µS cm^{-1})	Hardness (mg L^{-1} as $CaCO_3$)	Alkalinity (mg L^{-1} as $CaCO_3$)
1	Mean	7.95	21.5	0.4	7.78	418	96	90
	±SD	0.03	0.3	0.1	0.17	7		
	Minimum	7.91	21.2	0.3	7.6	409		
	Maximum	7.99	21.8	0.4	7.95	426		
2	Mean	7.89	21.6	0.4	7.79	420	101	94
	±SD	0.05	0.4	0.1	0.2	7		
	Minimum	7.83	21.2	0.3	7.58	412		
	Maximum	7.94	22	0.4	7.99	427		
3	Mean	7.91	21.6	0.4	7.77	423	101	94
	±SD	0.05	0.4	0	0.21	6		
	Minimum	7.85	21.2	0.3	7.55	417		
	Maximum	7.97	22	0.4	7.98	429		
4	Mean	7.83	21.4	0.5	7.74	427	96	90
	±SD	0.02	0.4	0.1	0.21	5		
	Minimum	7.79	21	0.4	7.53	421		
	Maximum	7.86	21.7	0.5	7.94	433		
5	Mean	7.87	21.5	0.5	7.9	431	101	94
	±SD	0.01	0.5	0.1	0.13	4		
	Minimum	7.85	21	0.4	7.76	425		
	Maximum	7.89	22	0.5	8.03	436		
6	Mean	7.9	21.5	0.5	7.88	435	101	94
	±SD	0.03	0.5	0.1	0.13	5		

	Minimum	7.86	21	0.4	7.74	429	101	94
	Maximum	7.94	22	0.5	8.02	440		
7	Mean	7.94	21.4	49.8	7.93	442		
	±SD	0.06	0.4	0.6	0.06	2	101	94
	Minimum	7.87	21	49	7.86	440		
	Maximum	8	21.8	50.7	8	445		
8	Mean	7.89	21.3	49.7	8.04	473		
	±SD	0.05	1	0.5	0.03	7	101	94
	Minimum	7.83	20.4	49	8	465		
	Maximum	7.94	22.2	50.5	8.08	480		
9	Mean	8.03	21.4	50.1	7.89	443		
	±SD	0.08	0.5	0.5	0.1	3	101	94
	Minimum	7.95	20.9	49.2	7.79	439		
	Maximum	8.11	21.9	50.9	7.99	448		
10	Mean	7.95	21.4	12.4	7.88	424		
	±SD	0.04	0.7	0.4	0.05	2	96	90
	Minimum	7.9	20.7	11.8	7.82	421		
	Maximum	7.98	22.1	12.9	7.94	427		
11	Mean	7.84	21.8	25	7.93	434		
	±SD	0.02	0.2	0.3	0.05	3	96	90
	Minimum	7.81	21.6	24.4	7.87	430		
	Maximum	7.86	22	25.5	7.99	437		
12	Mean	7.78	21.2	50.3	8	439		
	±SD	0.02	0.4	0.4	0.05	3	96	90
	Minimum	7.75	20.8	49.6	7.94	435		
	Maximum	7.8	21.6	50.8	8.06	443		
13	Mean	8.01	22	12.6	7.9	455		
	±SD	0.01	0.3	0.2	0.05	4	99	92
	Minimum	8	21.7	12.2	7.83	450		

	Maximum	8.01	22.3	12.9	7.96	460		
14	Mean	7.95	22.1	25.2	7.85	465	99	92
	±SD	0.03	0.6	0.3	0.07	7		
	Minimum	7.91	21.5	24.7	7.77	457		
	Maximum	7.98	22.6	25.7	7.92	472		
15	Mean	7.98	20.9	50.4	7.96	472	102	96
	±SD	0.02	0.2	0.3	0.06	7		
	Minimum	7.95	20.7	50	7.89	464		
	Maximum	8	21.1	50.8	8.03	480		
16	Mean	7.75	21.3	12.7	7.75	427	102	96
	±SD	0.04	0.6	0.2	0.03	4		
	Minimum	7.7	20.7	12.3	7.71	422		
	Maximum	7.8	21.8	13	7.79	431		
17	Mean	7.76	21.4	24.9	7.71	435	102	96
	±SD	0.05	0.1	0.2	0.02	3		
	Minimum	7.69	21.3	24.5	7.68	430		
	Maximum	7.81	21.4	25.2	7.74	439		
18	Mean	7.83	21.8	49.8	7.65	447	102	96
	±SD	0.04	0.5	0.3	0.03	9		
	Minimum	7.78	21.3	49.3	7.6	437		
	Maximum	7.88	22.3	50.2	7.69	456		
19	Mean	7.79	21.4	53.5	8.01	450	96	90
	±SD	0.02	0.1	0.3	0.05	5		
	Minimum	7.76	21.3	53.1	7.94	444		
	Maximum	7.82	21.5	54	8.07	456		
20	Mean	7.77	21.3	53.9	8.06	482	104	98
	±SD	0.03	0.1	0.3	0.05	4		
	Minimum	7.73	21.2	53.5	8	476		
	Maximum	7.8	21.4	54.3	8.12	487		

21	Mean	7.91	20.9	0.1	7.95	434		
	±SD	0.02	0.2	0.1	0.18	4	101	94
	Minimum	7.88	20.7	0.1	7.76	429		
	Maximum	7.95	21	0.2	8.13	440		
22	Mean	7.93	21.7	0.7	7.97	426		
	±SD	0.08	0.4	0.1	0.19	3	101	94
	Minimum	7.84	21.3	0.5	7.76	422		
	Maximum	8.01	22	0.8	8.16	429		
23	Mean	7.95	21.7	3	7.93	427		
	±SD	0.06	0.4	0.1	0.17	3	101	94
	Minimum	7.88	21.3	2.8	7.75	423		
	Maximum	8.01	22	3.1	8.1	430		
24	Mean	7.94	21.6	6.7	7.67	436		
	±SD	0.04	0.6	0.1	0.12	4	101	94
	Minimum	7.89	21	6.5	7.54	430		
	Maximum	7.98	22.2	6.8	7.79	441		
25	Mean	8.01	20.9	14	7.63	458		
	±SD	0.06	0.2	0.2	0.06	23	101	94
	Minimum	7.94	20.7	13.8	7.56	434		
	Maximum	8.07	21	14.3	7.69	480		
26	Mean	7.99	21.6	20	7.6	446		
	±SD	0.06	0.6	0.1	0.1	3	101	94
	Minimum	7.92	21	19.8	7.48	442		
	Maximum	8.06	22.2	20.1	7.7	450		
27	Mean	7.82	21.2	0.7	8.03	425		
	±SD	0.01	0.5	0.1	0.14	3	101	94
	Minimum	7.8	20.7	0.5	7.88	420		
	Maximum	7.84	21.6	0.9	8.17	429		
28	Mean	7.85	21.2	3.1	8.02	429		

	±SD	0.02	0.5	0.2	0.05	2	101	94
	Minimum	7.81	20.7	2.8	7.96	426		
	Maximum	7.87	21.6	3.3	8.08	432		
29	Mean	7.85	21.5	6.6	7.99	435	101	94
	±SD	0.05	0.6	0.2	0.14	3		
	Minimum	7.79	20.9	6.2	7.83	431		
	Maximum	7.9	22	6.9	8.14	439		
30	Mean	7.86	21.5	13.9	7.97	441	101	94
	±SD	0.06	0.6	0.2	0.17	3		
	Minimum	7.79	20.9	13.5	7.79	437		
	Maximum	7.93	22	14.2	8.14	445		
31	Mean	7.91	21.4	19.9	7.79	447	101	94
	±SD	0.05	0.4	0.3	0.02	4		
	Minimum	7.84	21	19.6	7.75	442		
	Maximum	7.96	21.8	20.6	7.82	453		
32	Mean	8.36	21.9	32.1	8.87	542	105	99
	±SD	0.02	0.2	7.6	0.04	5		
	Minimum	8.28	21.6	23	8.79	534		
	Maximum	8.39	22.3	55.3	9.09	551		
33	Mean	8	22.3	23	9.34	578	103	97
	±SD	0.11	0.6	8.4	0.12	9		
	Minimum	7.22	20.9	14.7	9.18	557		
	Maximum	8.05	23.2	53.7	9.68	591		
34	Mean	8.33	22.4	38.4	8.74	483	99	92
	±SD	0.02	0.5	8.3	0.1	4		
	Minimum	8.26	20.7	24.5	8.65	475		
	Maximum	8.35	22.9	61.1	9.08	491		

[a] NTU: Nephelometric turbidity units

[b] DO: Dissolved oxygen

Appendix table 6. Detailed physicochemical parameters for 31 batch experiments

TEST 1. NICKEL ONLY (207 µg L^{-1}) BATCH EXPERIMENT 29 JUNE-1 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.99	7.93	21.2	21.8	0.4	0.4	7.92	7.61	412	422		
2	7.97	7.91	21.2	21.8	0.4	0.3	7.95	7.62	409	423		
3	7.97	7.92	21.2	21.8	0.4	0.4	7.94	7.63	411	424		
4	7.96	7.91	21.2	21.8	0.3	0.3	7.94	7.60	413	426		
Time Mean	7.97	7.92	21.2	21.8	0.4	0.4	7.94	7.62	411	424		
Test Mean	7.95		21.5		0.4		7.78		418		96	90
±SD	0.03		0.3		0.1		0.17		7			
Minimum	7.91		21.2		0.3		7.60		409			
Maximum	7.99		21.8		0.4		7.95		426			

TEST 2. NICKEL ONLY (345 µg L^{-1}) BATCH EXPERIMENT 14-16 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.94	7.85	21.2	22.0	0.4	0.3	7.96	7.59	412	427		
2	7.93	7.84	21.2	22.0	0.3	0.3	7.98	7.62	414	426		
3	7.94	7.83	21.2	22.0	0.4	0.3	7.97	7.61	414	427		
4	7.92	7.84	21.2	22.0	0.4	0.4	7.99	7.58	416	424		
Time Mean	7.93	7.84	21.2	22.0	0.4	0.3	7.98	7.60	414	426		
Test Mean	7.89		21.6		0.4		7.79		420		101	94
±SD	0.05		0.4		0.1		0.20		7			
Minimum	7.83		21.2		0.3		7.58		412			
Maximum	7.94		22.0		0.4		7.99		427			

TEST 3. NICKEL ONLY (750 µg L^{-1}) BATCH EXPERIMENT 14-16 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.95	7.86	21.2	22.0	0.4	0.3	7.95	7.57	417	429		
2	7.96	7.86	21.2	22.0	0.4	0.4	7.97	7.61	417	428		
3	7.96	7.87	21.2	22.0	0.4	0.4	7.98	7.55	418	429		
4	7.97	7.85	21.2	22.0	0.4	0.4	7.97	7.56	419	427		
Time Mean	7.96	7.86	21.2	22.0	0.4	0.4	7.97	7.57	418	428		
Test Mean	7.91		21.6		0.4		7.77		423		101	94
±SD	0.05		0.4		0.0		0.21		6			
Minimum	7.85		21.2		0.3		7.55		417			
Maximum	7.97		22.0		0.4		7.98		429			

TEST 4. NICKEL ONLY (1516 µg L^{-1}) BATCH EXPERIMENT 6-8 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.85	7.79	21.0	21.7	0.5	0.4	7.94	7.53	424	430		
2	7.86	7.81	21.0	21.7	0.5	0.4	7.93	7.55	421	431		
3	7.84	7.82	21.0	21.7	0.4	0.4	7.93	7.55	423	431		
4	7.85	7.81	21.0	21.7	0.5	0.5	7.92	7.54	423	433		
Time Mean	7.85	7.81	21.0	21.7	0.5	0.4	7.93	7.54	423	431		
Test Mean	7.83		21.4		0.5		7.74		427		96	90
±SD	0.02		0.4		0.1		0.21		5			
Minimum	7.79		21.0		0.4		7.53		421			
Maximum	7.86		21.7		0.5		7.94		433			

TEST 5. NICKEL ONLY (3079 μg L^{-1}) BATCH EXPERIMENT 17-19 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(μS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.86	7.89	21.0	22.0	0.5	0.5	8.02	7.77	425	435		
2	7.85	7.87	21.0	22.0	0.5	0.5	8.03	7.76	426	433		
3	7.86	7.88	21.0	22.0	0.4	0.4	8.02	7.77	428	436		
4	7.86	7.88	21.0	22.0	0.4	0.4	8.01	7.78	427	434		
Time Mean	7.86	7.88	21.0	22.0	0.5	0.5	8.02	7.77	427	435		
Test Mean	7.87		21.5		0.5		7.90		431		101	94
±SD	0.01		0.5		0.1		0.13		4			
Minimum	7.85		21.0		0.4		7.76		425			
Maximum	7.89		22.0		0.5		8.03		436			

TEST 6. NICKEL ONLY (6153 μg L^{-1}) BATCH EXPERIMENT 17-19 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(μS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.87	7.93	21.0	22.0	0.4	0.4	7.99	7.74	430	439		
2	7.88	7.93	21.0	22.0	0.5	0.5	8.02	7.75	429	438		
3	7.87	7.94	21.0	22.0	0.5	0.5	7.98	7.77	429	439		
4	7.86	7.92	21.0	22.0	0.5	0.4	8.01	7.75	432	440		
Time Mean	7.87	7.93	21.0	22.0	0.5	0.5	8.00	7.75	430	439		
Test Mean	7.90		21.5		0.5		7.88		435		101	94
±SD	0.03		0.5		0.1		0.13		5			
Minimum	7.86		21.0		0.4		7.74		429			
Maximum	7.94		22.0		0.5		8.02		440			

TEST 7. WARDEN DITCH ONLY (50 NTU) BATCH EXPERIMENT 1-3 AUGUST 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	8.00	7.89	21.0	21.8	50.7	49.2	8.00	7.87	442	441		
2	7.99	7.88	21.0	21.8	50.1	49.0	7.98	7.86	443	441		
3	7.98	7.88	21.0	21.8	50.4	49.5	7.99	7.87	443	440		
4	7.99	7.87	21.0	21.8	49.7	49.4	7.97	7.88	445	442		
Time Mean	7.99	7.88	21.0	21.8	50.2	49.3	7.99	7.87	443	441		
Test Mean	7.94		21.4		49.8		7.93		442		101	94
±SD	0.06		0.4		0.6		0.06		2			
Minimum	7.87		21.0		49.0		7.86		440			
Maximum	8.00		21.8		50.7		8.00		445			

TEST 8. MONTMORILLONITE ONLY (50 NTU) BATCH EXPERIMENT 5-7 AUGUST 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.84	7.94	20.4	22.2	50.0	49.6	8.08	8.02	466	477		
2	7.83	7.93	20.4	22.2	49.6	49.0	8.06	8.00	467	480		
3	7.84	7.93	20.4	22.2	50.5	49.9	8.07	8.01	465	478		
4	7.85	7.94	20.4	22.2	49.4	49.4	8.05	8.00	468	479		
Time Mean	7.84	7.94	20.4	22.2	49.9	49.5	8.07	8.01	467	479		
Test Mean	7.89		21.3		49.7		8.04		473		101	94
±SD	0.05		1.0		0.5		0.03		7			
Minimum	7.83		20.4		49.0		8.00		465			
Maximum	7.94		22.2		50.5		8.08		480			

TEST 9. KAOLINITE ONLY (50 NTU) BATCH EXPERIMENT 3-5 AUGUST 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.96	8.09	20.9	21.9	50.4	49.7	7.96	7.81	439	445		
2	7.96	8.10	20.9	21.9	50.9	50.3	7.98	7.80	440	448		
3	7.95	8.10	20.9	21.9	50.2	49.8	7.99	7.81	441	446		
4	7.97	8.11	20.9	21.9	49.9	49.2	7.99	7.79	440	444		
Time Mean	7.96	8.10	20.9	21.9	50.4	49.8	7.98	7.80	440	446		
Test Mean	8.03		21.4		50.1		7.89		443		101	94
±SD	0.08		0.5		0.5		0.10		3			
Minimum	7.95		20.9		49.2		7.79		439			
Maximum	8.11		21.9		50.9		7.99		448			

TEST 10. Ni-SPIKED WARDEN DITCH (12.5 NTU) BATCH EXPERIMENT 22-24 JUNE 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.98	7.90	20.7	22.1	12.8	12.2	7.92	7.84	423	422		
2	7.98	7.92	20.7	22.1	12.2	11.8	7.94	7.83	427	424		
3	7.97	7.92	20.7	22.1	12.9	12.5	7.92	7.85	425	421		
4	7.98	7.91	20.7	22.1	12.6	12.2	7.91	7.82	424	423		
Time Mean	7.98	7.91	20.7	22.1	12.6	12.2	7.92	7.84	425	423		
Test Mean	7.95		21.4		12.4		7.88		424		96	90
±SD	0.04		0.7		0.4		0.05		2			
Minimum	7.90		20.7		11.8		7.82		421			
Maximum	7.98		22.1		12.9		7.94		427			

TEST 11. Ni-SPIKED WARDEN DITCH (25 NTU) BATCH EXPERIMENT 20-22 JUNE 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(μS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.82	7.86	21.6	22.0	25.0	24.7	7.97	7.87	431	435		
2	7.81	7.86	21.6	22.0	25.5	25.2	7.97	7.88	433	437		
3	7.82	7.85	21.6	22.0	24.8	24.4	7.98	7.87	430	437		
4	7.82	7.86	21.6	22.0	25.1	24.9	7.99	7.89	431	436		
Time Mean	7.82	7.86	21.6	22.0	25.1	24.8	7.98	7.88	431	436		
Test Mean	7.84		21.8		25.0		7.93		434		96	90
±SD	0.02		0.2		0.3		0.05		3			
Minimum	7.81		21.6		24.4		7.87		430			
Maximum	7.86		22.0		25.5		7.99		437			

TEST 12. Ni-SPIKED WARDEN DITCH (50 NTU) BATCH EXPERIMENT 18-20 JUNE 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(μS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.76	7.80	20.8	21.6	49.9	49.6	8.04	7.96	435	442		
2	7.75	7.79	20.8	21.6	50.4	50.2	8.06	7.95	436	441		
3	7.76	7.79	20.8	21.6	50.7	50.4	8.04	7.94	435	440		
4	7.77	7.79	20.8	21.6	50.8	50.1	8.05	7.96	437	443		
Time Mean	7.76	7.79	20.8	21.6	50.5	50.1	8.05	7.95	436	442		
Test Mean	7.78		21.2		50.3		8.00		439		96	90
±SD	0.02		0.4		0.4		0.05		3			
Minimum	7.75		20.8		49.6		7.94		435			
Maximum	7.80		21.6		50.8		8.06		443			

TEST 13. Ni-SPIKED MONTMORILLONITE (12.5 NTU) BATCH EXPERIMENT 29-31 MAY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	8.01	8.00	21.7	22.3	12.6	12.4	7.95	7.83	452	456		
2	8.01	8.00	21.7	22.3	12.7	12.5	7.96	7.86	450	457		
3	8.01	8.01	21.7	22.3	12.5	12.2	7.93	7.84	451	458		
4	8.00	8.00	21.7	22.3	12.9	12.6	7.92	7.87	453	460		
Time Mean	8.01	8.00	21.7	22.3	12.7	12.4	7.94	7.85	452	458		
Test Mean	8.01		22.0		12.6		7.90		455		99	92
±SD	0.01		0.3		0.2		0.05		4			
Minimum	8.00		21.7		12.2		7.83		450			
Maximum	8.01		22.3		12.9		7.96		460			

TEST 14. Ni-SPIKED MONTMORILLONITE (25 NTU) BATCH EXPERIMENT 26-28 MAY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.92	7.97	21.5	22.6	25.7	25.4	7.91	7.78	458	470		
2	7.91	7.96	21.5	22.6	25.0	24.7	7.89	7.77	459	472		
3	7.92	7.97	21.5	22.6	25.3	24.9	7.90	7.80	459	470		
4	7.93	7.98	21.5	22.6	25.5	25.1	7.92	7.79	457	471		
Time Mean	7.92	7.97	21.5	22.6	25.4	25.0	7.91	7.79	458	471		
Test Mean	7.95		22.1		25.2		7.85		465		99	92
±SD	0.03		0.6		0.3		0.07		7			
Minimum	7.91		21.5		24.7		7.77		457			
Maximum	7.98		22.6		25.7		7.92		472			

TEST 15. Ni-SPIKED MONTMORILLONITE (50 NTU) BATCH EXPERIMENT 6-8 MAY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.99	7.95	20.7	21.1	50.8	50.3	8.01	7.91	466	480		
2	8.00	7.97	20.7	21.1	50.3	50.0	8.02	7.89	465	477		
3	7.98	7.96	20.7	21.1	50.6	50.1	7.99	7.92	467	476		
4	7.99	7.96	20.7	21.1	50.7	50.2	8.03	7.93	464	479		
Time Mean	7.99	7.96	20.7	21.1	50.6	50.2	8.01	7.91	466	478		
Test Mean	7.98		20.9		50.4		7.96		472		102	96
±SD	0.02		0.2		0.3		0.06		7			
Minimum	7.95		20.7		50.0		7.89		464			
Maximum	8.00		21.1		50.8		8.03		480			

TEST 16. Ni-SPIKED KAOLINITE (12.5 NTU) BATCH EXPERIMENT 23-25 MAY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.70	7.79	20.7	21.8	12.9	12.6	7.75	7.71	423	431		
2	7.71	7.79	20.7	21.8	12.7	12.5	7.78	7.72	422	429		
3	7.71	7.78	20.7	21.8	12.6	12.3	7.77	7.72	423	430		
4	7.72	7.80	20.7	21.8	13.0	12.8	7.79	7.73	424	431		
Time Mean	7.71	7.79	20.7	21.8	12.8	12.6	7.77	7.72	423	430		
Test Mean	7.75		21.3		12.7		7.75		427		102	96
±SD	0.04		0.6		0.2		0.03		4			
Minimum	7.70		20.7		12.3		7.71		422			
Maximum	7.80		21.8		13.0		7.79		431			

TEST 17. Ni-SPIKED KAOLINITE (25 NTU) BATCH EXPERIMENT 21-23 MAY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.71	7.80	21.4	21.3	24.8	24.5	7.74	7.70	430	436		
2	7.69	7.80	21.4	21.3	25.0	24.6	7.73	7.69	432	438		
3	7.71	7.80	21.4	21.3	25.1	24.9	7.71	7.68	433	437		
4	7.72	7.81	21.4	21.3	25.2	24.8	7.72	7.70	431	439		
Time Mean	7.71	7.80	21.4	21.3	25.0	24.7	7.73	7.69	432	438		
Test Mean	7.76		21.4		24.9		7.71		435		102	96
±SD	0.05		0.1		0.2		0.02		3			
Minimum	7.69		21.3		24.5		7.68		430			
Maximum	7.81		21.4		25.2		7.74		439			

TEST 18. Ni-SPIKED KAOLINITE (50 NTU) BATCH EXPERIMENT 16-18 MAY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.78	7.87	21.3	22.3	49.9	49.5	7.69	7.62	437	456		
2	7.79	7.87	21.3	22.3	50.2	49.9	7.67	7.63	439	455		
3	7.80	7.88	21.3	22.3	50.0	49.6	7.66	7.61	438	454		
4	7.79	7.86	21.3	22.3	49.6	49.3	7.68	7.60	437	456		
Time Mean	7.79	7.87	21.3	22.3	49.9	49.6	7.68	7.62	438	455		
Test Mean	7.83		21.8		49.8		7.65		447		102	96
±SD	0.04		0.5		0.3		0.03		9			
Minimum	7.78		21.3		49.3		7.60		437			
Maximum	7.88		22.3		50.2		7.69		456			

TEST 19. Ni-SPIKED WARDEN DITCH (50 NTU) & ALDRICH HUMIC ACID BATCH EXPERIMENT 15-17 JUNE 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.82	7.77	21.5	21.3	54.0	53.6	8.06	7.97	446	454		
2	7.81	7.78	21.5	21.3	53.7	53.4	8.06	7.95	444	455		
3	7.81	7.77	21.5	21.3	53.4	53.1	8.04	7.94	445	453		
4	7.79	7.76	21.5	21.3	53.6	53.2	8.07	7.98	444	456		
Time Mean	7.81	7.77	21.5	21.3	53.7	53.3	8.06	7.96	445	455		
Test Mean	7.79		21.4		53.5		8.01		450		96	90
±SD	0.02		0.1		0.3		0.05		5			
Minimum	7.76		21.3		53.1		7.94		444			
Maximum	7.82		21.5		54.0		8.07		456			

TEST 20. Ni-SPIKED MONTMORILLONITE (50 NTU) & ALDRICH HUMIC ACID BATCH EXPERIMENT 11-13 JUNE 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.80	7.74	21.2	21.4	53.7	53.5	8.08	8.00	479	487		
2	7.79	7.74	21.2	21.4	53.9	53.6	8.11	8.02	476	485		
3	7.78	7.73	21.2	21.4	54.1	53.7	8.09	8.03	477	484		
4	7.79	7.75	21.2	21.4	54.3	54.0	8.12	8.01	479	487		
Time Mean	7.79	7.74	21.2	21.4	54.0	53.7	8.10	8.02	478	486		
Test Mean	7.77		21.3		53.9		8.06		482		104	98
±SD	0.03		0.1		0.3		0.05		4			
Minimum	7.73		21.2		53.5		8.00		476			
Maximum	7.80		21.4		54.3		8.12		487			

TEST 21. NICKEL (EC75) + ALDRICH HUMIC ACID (0 mg L^{-1}) BATCH EXPERIMENT 21-23 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.91	7.95	20.7	21.0	0.1	0.1	8.13	7.76	429	436		
2	7.89	7.93	20.7	21.0	0.2	0.2	8.13	7.77	430	440		
3	7.88	7.92	20.7	21.0	0.1	0.2	8.12	7.79	429	435		
4	7.89	7.92	20.7	21.0	0.1	0.1	8.11	7.80	431	438		
Time Mean	7.89	7.93	20.7	21.0	0.1	0.2	8.12	7.78	430	437		
Test Mean	7.91		20.9		0.1		7.95		434		101	94
±SD	0.02		0.2		0.1		0.18		4			
Minimum	7.88		20.7		0.1		7.76		429			
Maximum	7.95		21.0		0.2		8.13		440			

TEST 22. NICKEL (EC75) + ALDRICH HUMIC ACID (1 mg L^{-1}) BATCH EXPERIMENT 19-21 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	8.01	7.85	21.3	22.0	0.8	0.7	8.15	7.77	424	426		
2	8.00	7.84	21.3	22.0	0.6	0.5	8.12	7.81	423	429		
3	7.99	7.86	21.3	22.0	0.7	0.7	8.16	7.76	425	428		
4	8.00	7.88	21.3	22.0	0.7	0.6	8.13	7.82	422	429		
Time Mean	8.00	7.86	21.3	22.0	0.7	0.6	8.14	7.79	424	428		
Test Mean	7.93		21.7		0.7		7.97		426		101	94
±SD	0.08		0.4		0.1		0.19		3			
Minimum	7.84		21.3		0.5		7.76		422			
Maximum	8.01		22.0		0.8		8.16		429			

TEST 23. NICKEL (EC75) + ALDRICH HUMIC ACID (10 mg L^{-1}) BATCH EXPERIMENT 19-21 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.99	7.89	21.3	22.0	3.1	3.0	8.10	7.79	423	429		
2	8.00	7.88	21.3	22.0	3.0	2.9	8.08	7.75	424	429		
3	7.99	7.90	21.3	22.0	2.9	2.9	8.09	7.78	426	430		
4	8.01	7.90	21.3	22.0	3.0	2.8	8.07	7.76	425	428		
Time Mean	8.00	7.89	21.3	22.0	3.0	2.9	8.09	7.77	425	429		
Test Mean	7.95		21.7		3.0		7.93		427		101	94
±SD	0.06		0.4		0.1		0.17		3			
Minimum	7.88		21.3		2.8		7.75		423			
Maximum	8.01		22.0		3.1		8.10		430			

TEST 24. NICKEL (EC75) + ALDRICH HUMIC ACID (25 mg L^{-1}) BATCH EXPERIMENT 23-25 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.96	7.91	21.0	22.2	6.8	6.7	7.78	7.55	431	440		
2	7.97	7.90	21.0	22.2	6.5	6.6	7.79	7.57	432	441		
3	7.97	7.89	21.0	22.2	6.6	6.6	7.77	7.54	430	439		
4	7.98	7.91	21.0	22.2	6.7	6.7	7.78	7.54	433	438		
Time Mean	7.97	7.90	21.0	22.2	6.7	6.7	7.78	7.55	432	440		
Test Mean	7.94		21.6		6.7		7.67		436		101	94
±SD	0.04		0.6		0.1		0.12		4			
Minimum	7.89		21.0		6.5		7.54		430			
Maximum	7.98		22.2		6.8		7.79		441			

TEST 25. NICKEL (EC75) + ALDRICH HUMIC ACID (60 mg L^{-1}) BATCH EXPERIMENT 21-23 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	8.06	7.95	20.7	21.0	14.0	13.9	7.69	7.58	437	480		
2	8.07	7.96	20.7	21.0	14.3	14.1	7.68	7.59	438	477		
3	8.07	7.95	20.7	21.0	13.8	13.8	7.67	7.56	436	479		
4	8.05	7.94	20.7	21.0	13.9	13.8	7.69	7.57	434	479		
Time Mean	8.06	7.95	20.7	21.0	14.0	13.9	7.68	7.58	436	479		
Test Mean	8.01		20.9		14.0		7.63		458		101	94
±SD	0.06		0.2		0.2		0.06		23			
Minimum	7.94		20.7		13.8		7.56		434			
Maximum	8.07		21.0		14.3		7.69		480			

TEST 26. NICKEL (EC75) + ALDRICH HUMIC ACID (100 mg L^{-1}) BATCH EXPERIMENT 23-25 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	8.05	7.92	21.0	22.2	19.9	19.8	7.70	7.51	442	449		
2	8.06	7.94	21.0	22.2	20.0	20.0	7.67	7.48	443	449		
3	8.05	7.93	21.0	22.2	20.1	20.0	7.69	7.51	442	450		
4	8.04	7.93	21.0	22.2	19.9	19.9	7.68	7.52	444	448		
Time Mean	8.05	7.93	21.0	22.2	20.0	19.9	7.69	7.51	443	449		
Test Mean	7.99		21.6		20.0		7.60		446		101	94
±SD	0.06		0.6		0.1		0.10		3			
Minimum	7.92		21.0		19.8		7.48		442			
Maximum	8.06		22.2		20.1		7.70		450			

TEST 27. ALDRICH HUMIC ACID ONLY (1 mg L^{-1}) BATCH EXPERIMENT 28-30 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.84	7.80	20.7	21.6	0.7	0.6	8.15	7.91	424	429		
2	7.82	7.82	20.7	21.6	0.6	0.6	8.17	7.90	422	426		
3	7.83	7.81	20.7	21.6	0.6	0.5	8.16	7.88	420	428		
4	7.81	7.81	20.7	21.6	0.9	0.7	8.14	7.92	423	425		
Time Mean	7.83	7.81	20.7	21.6	0.7	0.6	8.16	7.90	422	427		
Test Mean	7.82		21.2		0.7		8.03		425		101	94
±SD	0.01		0.5		0.1		0.14		3			
Minimum	7.80		20.7		0.5		7.88		420			
Maximum	7.84		21.6		0.9		8.17		429			

TEST 28. ALDRICH HUMIC ACID ONLY (10 mg L^{-1}) BATCH EXPERIMENT 28-30 JULY 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.87	7.81	20.7	21.6	3.3	3.2	8.04	7.99	428	430		
2	7.86	7.84	20.7	21.6	2.9	2.8	8.08	7.97	427	432		
3	7.85	7.83	20.7	21.6	3.2	3.1	8.07	7.96	426	429		
4	7.87	7.84	20.7	21.6	3.1	3.0	8.05	7.98	427	430		
Time Mean	7.86	7.83	20.7	21.6	3.1	3.0	8.06	7.98	427	430		
Test Mean	7.85		21.2		3.1		8.02		429		101	94
±SD	0.02		0.5		0.2		0.05		2			
Minimum	7.81		20.7		2.8		7.96		426			
Maximum	7.87		21.6		3.3		8.08		432			

TEST 29. ALDRICH HUMIC ACID ONLY (25 mg L^{-1}) BATCH EXPERIMENT 30 JULY-1 AUGUST 2008

Replicate	**pH time 0 h**	**pH time 48 h**	**Temp time 0 h**	**Temp time 48 h**	**Turb time 0 h**	**Turb time 48 h**	**DO time 0 h**	**DO time 48 h**	**SC time 0 h**	**SC time 48 h**	**Hardness**	**Alkalinity**
	(units)		**(°C)**		**(NTU)**		**(mg L^{-1})**		**(µS cm^{-1})**		**(mg L^{-1} as $CaCO_3$)**	**(mg L^{-1} as $CaCO_3$)**
1	7.79	7.88	20.9	22.0	6.7	6.5	8.10	7.83	432	438		
2	7.81	7.90	20.9	22.0	6.9	6.2	8.12	7.85	431	436		
3	7.80	7.89	20.9	22.0	6.4	6.7	8.14	7.86	433	439		
4	7.82	7.89	20.9	22.0	6.6	6.4	8.11	7.87	432	437		
Time Mean	7.81	7.89	20.9	22.0	6.7	6.5	8.12	7.85	432	438		
Test Mean	7.85		21.5		6.6		7.99		435		101	94
±SD	0.05		0.6		0.2		0.14		3			
Minimum	7.79		20.9		6.2		7.83		431			
Maximum	7.90		22.0		6.9		8.14		439			

TEST 30. ALDRICH HUMIC ACID ONLY (60 mg L^{-1}) BATCH EXPERIMENT 30 JULY-1 AUGUST 2008

Replicate	**pH time 0 h**	**pH time 48 h**	**Temp time 0 h**	**Temp time 48 h**	**Turb time 0 h**	**Turb time 48 h**	**DO time 0 h**	**DO time 48 h**	**SC time 0 h**	**SC time 48 h**	**Hardness**	**Alkalinity**
	(units)		**(°C)**		**(NTU)**		**(mg L^{-1})**		**(µS cm^{-1})**		**(mg L^{-1} as $CaCO_3$)**	**(mg L^{-1} as $CaCO_3$)**
1	7.80	7.89	20.9	22.0	14.2	14.0	8.13	7.81	438	443		
2	7.80	7.91	20.9	22.0	14.0	13.9	8.11	7.82	439	441		
3	7.79	7.91	20.9	22.0	13.9	13.8	8.12	7.81	437	442		
4	7.81	7.93	20.9	22.0	13.6	13.5	8.14	7.79	439	445		
Time Mean	7.80	7.91	20.9	22.0	13.9	13.8	8.13	7.81	438	443		
Test Mean	7.86		21.5		13.9		7.97		441		101	94
±SD	0.06		0.6		0.2		0.17		3			
Minimum	7.79		20.9		13.5		7.79		437			
Maximum	7.93		22.0		14.2		8.14		445			

TEST 31. ALDRICH HUMIC ACID ONLY (100 mg L^{-1}) BATCH EXPERIMENT 1-3 AUGUST 2008

Replicate	pH time 0 h	pH time 48 h	Temp time 0 h	Temp time 48 h	Turb time 0 h	Turb time 48 h	DO time 0 h	DO time 48 h	SC time 0 h	SC time 48 h	Hardness	Alkalinity
	(units)		(°C)		(NTU)		(mg L^{-1})		(µS cm^{-1})		(mg L^{-1} as $CaCO_3$)	(mg L^{-1} as $CaCO_3$)
1	7.94	7.84	21.0	21.8	19.9	19.9	7.77	7.78	444	453		
2	7.96	7.86	21.0	21.8	19.8	19.7	7.79	7.82	443	450		
3	7.95	7.86	21.0	21.8	20.6	19.6	7.78	7.81	443	449		
4	7.96	7.87	21.0	21.8	20.2	19.7	7.75	7.80	442	448		
Time Mean	7.95	7.86	21.0	21.8	20.1	19.7	7.77	7.80	443	450		
Test Mean	7.91		21.4		19.9		7.79		447		101	94
±SD	0.05		0.4		0.3		0.02		4			
Minimum	7.84		21.0		19.6		7.75		442			
Maximum	7.96		21.8		20.6		7.82		453			

Appendix table 7. Detailed physicochemical parameters for 3 SRF experiments

Fifteen-Minute Interval Sonde Physicochemical Data for SRF Experiments

TEST 32. Ni-SPIKED WARDEN DITCH (50 NTU) SRF EXPERIMENT 19-21 JUNE 2008

Date	Temperature (°C)	Specific Conductance ($\mu S\ cm^{-1}$)	pH (units)	Turbidity (NTU)	DO ($mg\ L^{-1}$)
6/19/2008	21.6	534	8.28	55.3	8.92
6/19/2008	21.6	534	8.30	55.0	8.95
6/19/2008	21.6	534	8.31	54.8	8.96
6/19/2008	21.6	534	8.32	54.2	8.96
6/19/2008	21.7	534	8.33	53.7	8.95
6/19/2008	21.7	534	8.34	50.1	8.94
6/19/2008	21.7	534	8.35	49.1	8.94
6/19/2008	21.7	534	8.35	48.7	8.93
6/19/2008	21.7	534	8.36	48.7	8.93
6/19/2008	21.7	534	8.36	47.2	8.92
6/19/2008	21.8	534	8.37	46.6	8.91
6/19/2008	21.8	534	8.37	45.6	8.90
6/19/2008	21.8	534	8.37	45.2	8.90
6/19/2008	21.8	534	8.38	44.9	8.89
6/19/2008	21.8	534	8.38	44.2	8.89
6/19/2008	21.8	534	8.38	43.7	8.89
6/19/2008	21.8	534	8.38	43.3	8.89
6/19/2008	21.8	535	8.38	43.7	8.88
6/19/2008	21.8	535	8.38	43.6	8.88
6/19/2008	21.8	535	8.38	44.3	8.88
6/19/2008	21.9	535	8.38	42.7	8.88

6/19/2008	21.9	535	8.38	43.2	8.88
6/19/2008	21.8	535	8.38	43.2	8.89
6/19/2008	21.8	535	8.39	41.6	8.88
6/19/2008	21.8	535	8.38	41.4	8.89
6/19/2008	21.8	535	8.38	41.1	8.90
6/19/2008	21.8	535	8.38	40.8	8.90
6/19/2008	21.8	535	8.38	40.6	8.90
6/20/2008	21.8	535	8.38	40.2	8.90
6/20/2008	21.8	536	8.38	39.9	8.90
6/20/2008	21.8	536	8.38	40.0	8.90
6/20/2008	21.8	536	8.38	39.1	8.90
6/20/2008	21.8	536	8.38	39.3	8.90
6/20/2008	21.8	536	8.38	38.6	8.90
6/20/2008	21.7	536	8.38	39.6	8.90
6/20/2008	21.7	536	8.38	39.0	8.90
6/20/2008	21.7	536	8.38	38.3	8.90
6/20/2008	21.7	536	8.37	38.8	8.90
6/20/2008	21.7	536	8.37	38.5	8.90
6/20/2008	21.7	536	8.37	38.4	8.90
6/20/2008	21.7	536	8.37	38.5	8.91
6/20/2008	21.7	536	8.37	37.6	8.91
6/20/2008	21.7	536	8.37	37.6	8.92
6/20/2008	21.7	536	8.36	37.6	8.92
6/20/2008	21.7	536	8.36	36.7	8.91
6/20/2008	21.7	537	8.36	38.2	8.91
6/20/2008	21.7	537	8.36	36.7	8.91
6/20/2008	21.7	537	8.36	37.5	8.91
6/20/2008	21.7	537	8.36	36.5	8.92
6/20/2008	21.7	537	8.36	36.3	8.92

6/20/2008	21.6	537	8.36	37.7	8.92
6/20/2008	21.6	537	8.35	36.0	8.92
6/20/2008	21.6	537	8.35	36.1	8.92
6/20/2008	21.7	537	8.35	36.0	8.91
6/20/2008	21.7	537	8.35	36.1	8.92
6/20/2008	21.7	538	8.35	35.4	8.91
6/20/2008	21.7	538	8.35	35.2	8.91
6/20/2008	21.7	538	8.34	34.9	8.91
6/20/2008	21.7	538	8.34	35.2	8.91
6/20/2008	21.8	538	8.34	34.2	8.90
6/20/2008	21.8	538	8.34	34.4	8.89
6/20/2008	21.8	538	8.34	35.0	8.90
6/20/2008	21.8	538	8.34	34.1	8.89
6/20/2008	21.8	538	8.34	34.1	8.89
6/20/2008	21.9	538	8.34	33.9	8.88
6/20/2008	21.9	539	8.34	34.6	8.88
6/20/2008	21.9	539	8.34	34.4	8.88
6/20/2008	21.9	539	8.34	33.5	8.87
6/20/2008	21.9	539	8.34	32.7	8.87
6/20/2008	22.0	539	8.34	33.9	8.86
6/20/2008	22.0	539	8.34	32.9	8.86
6/20/2008	22.0	539	8.34	32.7	8.86
6/20/2008	22.0	539	8.34	32.6	8.86
6/20/2008	22.0	539	8.34	32.2	8.85
6/20/2008	22.0	540	8.34	33.2	8.85
6/20/2008	22.0	540	8.34	32.1	8.86
6/20/2008	22.0	540	8.35	32.0	8.85
6/20/2008	22.1	540	8.35	31.3	8.85
6/20/2008	22.1	540	8.35	31.0	8.84

6/20/2008	22.1	540	8.35	31.1	8.85
6/20/2008	22.1	540	8.35	31.3	9.09
6/20/2008	22.1	540	8.35	31.0	8.84
6/20/2008	22.1	540	8.35	30.9	8.84
6/20/2008	22.1	540	8.35	31.1	8.83
6/20/2008	22.1	541	8.35	29.9	8.84
6/20/2008	22.1	541	8.36	29.9	8.83
6/20/2008	22.1	541	8.36	31.1	8.84
6/20/2008	22.1	541	8.36	30.0	8.84
6/20/2008	22.1	541	8.36	30.6	8.84
6/20/2008	22.1	541	8.36	30.0	8.84
6/20/2008	22.1	541	8.36	30.3	8.84
6/20/2008	22.1	541	8.36	29.4	8.84
6/20/2008	22.1	541	8.36	29.9	8.84
6/20/2008	22.1	541	8.36	29.2	8.84
6/20/2008	22.1	541	8.36	29.4	8.84
6/20/2008	22.1	542	8.36	29.7	8.84
6/20/2008	22.1	542	8.37	29.0	8.84
6/20/2008	22.1	542	8.37	29.4	8.84
6/20/2008	22.1	542	8.37	29.4	8.84
6/20/2008	22.1	542	8.37	28.7	8.84
6/20/2008	22.1	542	8.37	28.8	8.84
6/20/2008	22.1	542	8.37	28.7	8.83
6/20/2008	22.1	543	8.37	28.5	8.84
6/20/2008	22.1	543	8.37	29.3	8.83
6/20/2008	22.1	543	8.37	28.2	8.83
6/20/2008	22.1	543	8.37	28.3	8.83
6/20/2008	22.1	543	8.37	28.3	8.82
6/20/2008	22.1	543	8.37	28.3	8.83

6/20/2008	22.1	543	8.37	27.8	8.83
6/20/2008	22.1	543	8.37	28.1	8.84
6/20/2008	22.1	543	8.37	27.8	8.84
6/20/2008	22.1	543	8.37	28.0	8.84
6/20/2008	22.1	543	8.38	27.8	8.84
6/20/2008	22.1	544	8.38	27.9	8.84
6/20/2008	22.1	544	8.38	27.5	8.84
6/20/2008	22.1	544	8.38	28.6	8.84
6/20/2008	22.1	544	8.38	28.0	8.85
6/20/2008	22.1	544	8.38	27.6	8.84
6/20/2008	22.1	544	8.38	27.7	8.85
6/20/2008	22.0	544	8.38	27.9	8.85
6/20/2008	22.0	544	8.38	27.8	8.86
6/20/2008	22.0	544	8.37	27.5	8.87
6/20/2008	22.0	544	8.38	27.1	8.87
6/20/2008	22.0	544	8.37	27.4	8.87
6/21/2008	22.0	544	8.38	27.2	8.87
6/21/2008	21.9	544	8.37	27.5	8.88
6/21/2008	21.9	545	8.37	26.9	8.89
6/21/2008	21.9	545	8.37	27.7	8.88
6/21/2008	21.9	545	8.37	27.2	8.89
6/21/2008	21.9	545	8.37	26.9	8.89
6/21/2008	21.9	545	8.37	26.8	8.89
6/21/2008	21.9	545	8.37	27.1	8.89
6/21/2008	21.9	545	8.37	26.7	8.89
6/21/2008	21.9	545	8.37	26.6	8.89
6/21/2008	21.9	545	8.37	26.8	8.89
6/21/2008	21.9	545	8.37	26.6	8.89
6/21/2008	21.9	545	8.37	26.9	8.90

6/21/2008	21.9	545	8.36	26.4	8.90
6/21/2008	21.9	546	8.36	26.2	8.89
6/21/2008	21.8	546	8.36	26.6	8.90
6/21/2008	21.8	546	8.36	26.4	8.90
6/21/2008	21.8	546	8.36	27.5	8.91
6/21/2008	21.8	546	8.36	26.0	8.90
6/21/2008	21.8	546	8.36	26.6	8.91
6/21/2008	21.8	546	8.35	26.4	8.91
6/21/2008	21.8	546	8.35	26.9	8.90
6/21/2008	21.8	546	8.35	26.4	8.91
6/21/2008	21.8	546	8.35	26.1	8.91
6/21/2008	21.8	546	8.35	26.2	8.91
6/21/2008	21.9	546	8.35	26.1	8.91
6/21/2008	21.9	547	8.35	26.3	8.90
6/21/2008	21.9	547	8.35	25.5	8.89
6/21/2008	21.9	547	8.35	26.3	8.89
6/21/2008	21.9	547	8.35	25.8	8.90
6/21/2008	21.9	547	8.35	25.6	8.90
6/21/2008	22.0	547	8.35	26.1	8.88
6/21/2008	22.0	547	8.35	25.6	8.88
6/21/2008	22.0	547	8.35	25.8	8.87
6/21/2008	22.0	547	8.35	24.9	8.87
6/21/2008	22.0	547	8.35	25.3	8.86
6/21/2008	22.1	548	8.35	25.7	8.86
6/21/2008	22.1	548	8.35	25.2	8.86
6/21/2008	22.1	548	8.35	24.9	8.86
6/21/2008	22.1	548	8.35	25.3	8.85
6/21/2008	22.1	548	8.35	24.9	8.85
6/21/2008	22.1	548	8.35	25.0	8.85

6/21/2008	22.2	548	8.36	24.7	8.84
6/21/2008	22.2	548	8.36	24.7	8.84
6/21/2008	22.2	549	8.36	24.5	8.84
6/21/2008	22.2	549	8.36	24.7	8.83
6/21/2008	22.2	549	8.36	24.6	8.83
6/21/2008	22.2	549	8.36	24.2	8.83
6/21/2008	22.2	549	8.36	24.9	8.83
6/21/2008	22.2	549	8.37	25.2	8.82
6/21/2008	22.2	549	8.37	24.4	8.82
6/21/2008	22.3	549	8.37	23.9	8.82
6/21/2008	22.3	549	8.37	24.0	8.82
6/21/2008	22.3	549	8.37	24.1	8.81
6/21/2008	22.3	549	8.37	24.1	8.81
6/21/2008	22.3	550	8.37	23.9	8.82
6/21/2008	22.3	549	8.37	23.6	8.82
6/21/2008	22.3	550	8.38	23.8	8.80
6/21/2008	22.3	550	8.38	24.0	8.80
6/21/2008	22.3	550	8.38	23.3	8.80
6/21/2008	22.3	550	8.38	23.4	8.80
6/21/2008	22.3	550	8.38	23.7	8.80
6/21/2008	22.3	550	8.38	23.4	8.79
6/21/2008	22.3	550	8.38	23.0	8.80
6/21/2008	22.3	550	8.38	23.2	8.80
6/21/2008	22.3	550	8.39	23.3	8.79
6/21/2008	22.3	551	8.39	23.0	8.80
6/21/2008	22.3	551	8.39	23.4	8.80

Ni-SPIKED WARDEN DITCH (50 NTU) SRF EXPERIMENT 19-21 JUNE 2008

Statistic	Temperature (°C)	Specific Conductance (μS cm^{-1})	pH (units)	Turbidity (NTU)	DO (mg L^{-1})
Mean	21.9	542	8.36	32.1	8.87
±SD	0.2	5	0.02	7.6	0.04
Minimum	21.6	534	8.28	23.0	8.79
Maximum	22.3	551	8.39	55.3	9.09

Hardness (mg L^{-1} as $CaCO_3$)	Alkalinity (mg L^{-1} as $CaCO_3$)
105	99

Fifteen-Minute Interval Sonde Physicochemical Data for SRF Experiments

TEST 33. Ni-SPIKED MONTMORILLONITE (50 NTU) SRF EXPERIMENT 13-15 MAY 2008

Date	Temperature (°C)	Specific Conductance (µS cm^{-1})	pH (units)	Turbidity (NTU)	DO (mg L^{-1})
5/13/2008	20.9	557	7.22	53.7	9.41
5/13/2008	20.9	559	7.23	52.8	9.68
5/13/2008	20.9	559	7.45	51.9	9.65
5/13/2008	20.9	560	7.59	49.9	9.65
5/13/2008	20.9	560	7.69	47.0	9.64
5/13/2008	20.9	560	7.75	46.6	9.63
5/13/2008	21.0	560	7.80	44.3	9.63
5/13/2008	21.0	561	7.83	42.2	9.62
5/13/2008	21.0	561	7.86	43.5	9.62
5/13/2008	21.0	561	7.88	42.1	9.61
5/13/2008	21.1	561	7.90	40.3	9.60
5/13/2008	21.1	562	7.92	38.5	9.60
5/13/2008	21.1	562	7.93	38.3	9.59
5/13/2008	21.2	562	7.94	38.3	9.59
5/13/2008	21.2	562	7.95	37.5	9.58
5/13/2008	21.2	562	7.96	35.5	9.58
5/13/2008	21.3	563	7.96	36.1	9.57
5/13/2008	21.3	563	7.97	35.7	9.56
5/13/2008	21.4	563	7.97	35.2	9.55
5/13/2008	21.4	564	7.98	34.7	9.53
5/13/2008	21.5	564	7.98	33.4	9.52
5/13/2008	21.5	564	7.99	33.8	9.51
5/13/2008	21.6	564	7.99	33.0	9.49

5/13/2008	21.6	565	7.99	32.5	9.48
5/13/2008	21.7	565	7.99	31.9	9.47
5/13/2008	21.7	565	7.99	31.9	9.46
5/13/2008	21.7	565	8.00	30.8	9.46
5/13/2008	21.8	566	8.00	31.0	9.44
5/13/2008	21.8	566	8.00	30.7	9.43
5/13/2008	21.9	566	8.00	31.1	9.42
5/13/2008	21.9	566	8.00	29.6	9.41
5/13/2008	21.9	567	8.00	29.3	9.40
5/13/2008	22.0	567	8.00	33.8	9.39
5/13/2008	22.0	567	8.01	29.4	9.39
5/13/2008	22.0	567	8.01	29.7	9.38
5/13/2008	22.0	568	8.01	29.1	9.38
5/13/2008	22.1	568	8.01	29.4	9.36
5/13/2008	22.1	568	8.01	28.8	9.36
5/13/2008	22.1	568	8.01	28.7	9.35
5/13/2008	22.2	569	8.01	29.9	9.34
5/13/2008	22.2	569	8.01	28.5	9.33
5/13/2008	22.2	569	8.02	28.8	9.33
5/13/2008	22.2	569	8.02	27.7	9.33
5/13/2008	22.3	570	8.02	27.3	9.32
5/13/2008	22.3	570	8.02	27.5	9.31
5/13/2008	22.3	570	8.02	27.1	9.31
5/13/2008	22.3	570	8.02	26.8	9.30
5/13/2008	22.4	571	8.02	26.7	9.29
5/13/2008	22.4	571	8.02	26.4	9.29
5/13/2008	22.4	571	8.02	26.3	9.30
5/13/2008	22.4	571	8.02	26.7	9.29
5/13/2008	22.4	572	8.03	26.2	9.28

5/13/2008	22.4	572	8.03	26.0	9.28
5/13/2008	22.4	572	8.03	25.8	9.28
5/13/2008	22.4	572	8.03	25.5	9.28
5/13/2008	22.4	572	8.03	25.4	9.28
5/13/2008	22.4	573	8.03	25.2	9.29
5/13/2008	22.4	573	8.03	25.1	9.29
5/13/2008	22.4	573	8.03	24.5	9.29
5/13/2008	22.4	573	8.03	25.0	9.28
5/13/2008	22.4	573	8.03	24.8	9.29
5/13/2008	22.3	574	8.03	24.5	9.29
5/13/2008	22.3	574	8.03	24.0	9.30
5/13/2008	22.3	574	8.03	23.9	9.31
5/13/2008	22.3	574	8.03	23.8	9.31
5/14/2008	22.3	574	8.03	23.9	9.31
5/14/2008	22.2	574	8.03	23.5	9.32
5/14/2008	22.2	575	8.03	23.2	9.32
5/14/2008	22.2	575	8.03	23.8	9.32
5/14/2008	22.2	575	8.03	22.8	9.32
5/14/2008	22.1	575	8.03	23.0	9.33
5/14/2008	22.1	575	8.03	23.0	9.33
5/14/2008	22.1	575	8.02	22.7	9.33
5/14/2008	22.1	575	8.02	22.1	9.35
5/14/2008	22.1	575	8.02	22.2	9.34
5/14/2008	22.0	576	8.02	22.4	9.35
5/14/2008	22.0	576	8.02	22.0	9.35
5/14/2008	22.0	576	8.02	21.7	9.36
5/14/2008	22.0	576	8.02	21.9	9.36
5/14/2008	22.0	576	8.02	22.0	9.36
5/14/2008	21.9	576	8.02	21.8	9.37

5/14/2008	21.9	577	8.02	21.4	9.37
5/14/2008	21.9	577	8.02	21.3	9.37
5/14/2008	21.9	577	8.02	21.1	9.37
5/14/2008	21.9	577	8.02	21.2	9.37
5/14/2008	21.8	577	8.02	21.0	9.38
5/14/2008	21.8	577	8.02	20.9	9.39
5/14/2008	21.8	578	8.02	21.3	9.39
5/14/2008	21.8	578	8.02	20.8	9.40
5/14/2008	21.8	578	8.02	20.5	9.40
5/14/2008	21.8	578	8.02	20.4	9.40
5/14/2008	21.8	578	8.02	20.6	9.41
5/14/2008	21.8	578	8.02	20.5	9.41
5/14/2008	21.8	578	8.02	20.2	9.41
5/14/2008	21.8	579	8.02	20.0	9.41
5/14/2008	21.8	579	8.02	19.9	9.41
5/14/2008	21.8	579	8.01	20.2	9.41
5/14/2008	21.8	579	8.01	20.0	9.41
5/14/2008	21.8	579	8.01	20.2	9.40
5/14/2008	21.8	579	8.01	20.1	9.40
5/14/2008	21.8	579	8.01	19.7	9.41
5/14/2008	21.8	580	8.01	19.7	9.41
5/14/2008	21.8	580	8.01	19.8	9.41
5/14/2008	21.9	580	8.01	19.6	9.41
5/14/2008	21.9	580	8.01	19.2	9.41
5/14/2008	21.9	580	8.01	19.5	9.40
5/14/2008	21.9	580	8.01	19.2	9.40
5/14/2008	21.9	580	8.01	19.1	9.40
5/14/2008	21.9	581	8.00	19.3	9.39
5/14/2008	21.9	581	8.00	18.8	9.39

5/14/2008	22.0	581	8.00	18.9	9.39
5/14/2008	22.0	581	8.00	18.8	9.39
5/14/2008	22.0	581	8.00	18.7	9.38
5/14/2008	22.0	581	8.00	19.0	9.38
5/14/2008	22.1	582	8.00	18.5	9.37
5/14/2008	22.1	582	8.00	18.5	9.37
5/14/2008	22.1	582	8.00	18.8	9.36
5/14/2008	22.1	582	8.00	18.3	9.36
5/14/2008	22.1	582	8.01	18.4	9.36
5/14/2008	22.2	582	8.01	18.6	9.36
5/14/2008	22.2	582	8.02	18.2	9.35
5/14/2008	22.2	583	8.02	18.2	9.35
5/14/2008	22.2	583	8.02	18.3	9.35
5/14/2008	22.2	583	8.03	18.3	9.35
5/14/2008	22.2	583	8.03	17.8	9.34
5/14/2008	22.3	583	8.03	18.1	9.33
5/14/2008	22.3	583	8.03	17.8	9.33
5/14/2008	22.3	583	8.03	17.9	9.32
5/14/2008	22.3	584	8.03	17.8	9.32
5/14/2008	22.4	584	8.03	17.7	9.32
5/14/2008	22.4	584	8.03	17.7	9.31
5/14/2008	22.5	584	8.03	17.7	9.30
5/14/2008	22.5	584	8.03	17.4	9.29
5/14/2008	22.6	584	8.03	17.5	9.28
5/14/2008	22.6	584	8.03	17.5	9.27
5/14/2008	22.6	584	8.03	17.3	9.26
5/14/2008	22.7	585	8.03	17.3	9.26
5/14/2008	22.7	585	8.03	17.2	9.25
5/14/2008	22.8	585	8.03	17.0	9.24

5/14/2008	22.8	585	8.03	17.2	9.24
5/14/2008	22.8	585	8.03	17.0	9.23
5/14/2008	22.9	585	8.03	16.9	9.23
5/14/2008	22.9	586	8.04	16.8	9.23
5/14/2008	22.9	585	8.04	16.9	9.22
5/14/2008	22.9	586	8.04	16.8	9.22
5/14/2008	23.0	586	8.04	16.9	9.21
5/14/2008	23.0	586	8.04	17.0	9.20
5/14/2008	23.0	586	8.04	16.6	9.20
5/14/2008	23.1	586	8.04	16.5	9.20
5/14/2008	23.1	587	8.04	16.6	9.20
5/14/2008	23.1	587	8.04	16.4	9.19
5/14/2008	23.1	587	8.04	16.4	9.19
5/14/2008	23.1	587	8.04	16.4	9.19
5/14/2008	23.2	587	8.04	16.4	9.18
5/14/2008	23.2	587	8.04	16.3	9.18
5/14/2008	23.2	587	8.04	16.2	9.18
5/14/2008	23.2	587	8.04	16.2	9.18
5/14/2008	23.2	587	8.04	16.1	9.19
5/14/2008	23.2	588	8.04	16.2	9.19
5/14/2008	23.2	588	8.04	16.0	9.19
5/14/2008	23.2	588	8.04	16.0	9.18
5/15/2008	23.2	588	8.04	16.1	9.19
5/15/2008	23.2	588	8.05	16.0	9.19
5/15/2008	23.2	588	8.05	15.8	9.19
5/15/2008	23.2	588	8.05	15.8	9.20
5/15/2008	23.2	588	8.05	15.7	9.20
5/15/2008	23.2	588	8.05	15.8	9.20
5/15/2008	23.2	589	8.05	15.7	9.20

5/15/2008	23.2	588	8.05	15.6	9.20
5/15/2008	23.2	589	8.05	15.7	9.20
5/15/2008	23.2	589	8.05	15.5	9.20
5/15/2008	23.1	589	8.05	15.5	9.21
5/15/2008	23.1	589	8.05	15.5	9.21
5/15/2008	23.1	589	8.05	15.4	9.21
5/15/2008	23.1	589	8.05	15.4	9.21
5/15/2008	23.1	589	8.05	15.5	9.21
5/15/2008	23.1	589	8.05	15.3	9.22
5/15/2008	23.1	589	8.05	15.4	9.22
5/15/2008	23.1	589	8.05	15.2	9.22
5/15/2008	23.1	590	8.05	15.3	9.22
5/15/2008	23.1	590	8.05	15.3	9.23
5/15/2008	23.1	590	8.05	15.1	9.23
5/15/2008	23.1	590	8.05	15.1	9.23
5/15/2008	23.0	590	8.05	15.2	9.23
5/15/2008	23.0	590	8.05	15.1	9.24
5/15/2008	23.0	590	8.05	15.0	9.24
5/15/2008	23.0	590	8.05	15.0	9.24
5/15/2008	23.0	590	8.05	15.0	9.25
5/15/2008	23.0	590	8.05	15.0	9.25
5/15/2008	23.1	591	8.05	14.9	9.25
5/15/2008	23.1	591	8.05	14.7	9.24
5/15/2008	23.1	591	8.05	15.0	9.25

Ni-SPIKED MONTMORILLONITE (50 NTU) SRF EXPERIMENT 13-15 MAY 2008

Statistic	Temperature (°C)	Specific Conductance (μS cm^{-1})	pH (units)	Turbidity (NTU)	DO (mg L^{-1})
Mean	22.3	578	8.00	23.0	9.34
±SD	0.6	9	0.11	8.4	0.12
Minimum	20.9	557	7.22	14.7	9.18
Maximum	23.2	591	8.05	53.7	9.68

Hardness (mg L^{-1} as $CaCO_3$)	Alkalinity (mg L^{-1} as $CaCO_3$)
103	97

Fifteen-Minute Interval Sonde Physicochemical Data for SRF Experiments

TEST 34. Ni-SPIKED KAOLINITE (50 NTU) SRF EXPERIMENT 25-27 MAY 2008

Date	Temperature (°C)	Specific Conductance ($\mu S\ cm^{-1}$)	pH (units)	Turbidity (NTU)	DO ($mg\ L^{-1}$)
5/25/2008	20.7	476	8.26	54.1	9.03
5/25/2008	20.8	475	8.26	52.8	9.07
5/25/2008	20.8	476	8.27	49.7	9.08
5/25/2008	20.9	476	8.27	61.1	9.08
5/25/2008	20.9	476	8.28	56.1	9.07
5/25/2008	20.9	476	8.28	55.7	9.06
5/25/2008	21.0	476	8.28	47.8	9.04
5/25/2008	21.0	476	8.28	52.0	9.03
5/25/2008	21.1	476	8.29	47.2	9.03
5/25/2008	21.1	476	8.29	58.4	9.01
5/25/2008	21.2	476	8.29	56.4	9.00
5/25/2008	21.2	476	8.29	53.7	8.98
5/25/2008	21.3	476	8.29	55.2	8.98
5/25/2008	21.3	477	8.30	54.6	8.97
5/25/2008	21.4	477	8.30	52.7	8.95
5/25/2008	21.4	477	8.30	46.6	8.95
5/25/2008	21.5	477	8.30	51.6	8.93
5/25/2008	21.6	477	8.30	54.5	8.92
5/25/2008	21.6	477	8.31	50.2	8.90
5/25/2008	21.6	477	8.31	47.1	8.90
5/25/2008	21.7	477	8.31	52.6	8.89
5/25/2008	21.7	477	8.31	45.8	8.88
5/25/2008	21.8	477	8.31	50.9	8.87

5/25/2008	21.8	477	8.31	54.0	8.86
5/25/2008	21.8	477	8.31	49.3	8.85
5/25/2008	21.9	477	8.31	44.4	8.85
5/25/2008	21.9	478	8.32	44.1	8.84
5/25/2008	21.9	478	8.32	47.3	8.83
5/25/2008	22.0	478	8.32	46.2	8.83
5/25/2008	22.0	478	8.32	42.5	8.82
5/25/2008	22.0	478	8.32	44.3	8.82
5/25/2008	22.1	478	8.32	43.0	8.81
5/25/2008	22.1	478	8.32	50.6	8.80
5/25/2008	22.1	478	8.32	49.9	8.79
5/25/2008	22.2	478	8.33	46.2	8.78
5/25/2008	22.2	479	8.33	45.9	8.78
5/25/2008	22.2	479	8.33	44.8	8.77
5/25/2008	22.2	479	8.33	45.7	8.77
5/25/2008	22.2	479	8.33	44.6	8.77
5/25/2008	22.3	479	8.33	41.1	8.76
5/25/2008	22.3	479	8.33	50.4	8.76
5/25/2008	22.3	479	8.33	42.1	8.75
5/25/2008	22.3	479	8.33	41.9	8.75
5/25/2008	22.3	479	8.33	46.6	8.74
5/25/2008	22.4	479	8.33	43.0	8.74
5/25/2008	22.4	479	8.33	40.8	8.73
5/25/2008	22.4	480	8.33	50.2	8.73
5/25/2008	22.4	480	8.33	40.1	8.72
5/25/2008	22.4	480	8.33	44.0	8.72
5/25/2008	22.4	480	8.33	43.7	8.71
5/25/2008	22.5	480	8.33	46.2	8.71
5/25/2008	22.5	480	8.33	39.0	8.71

5/25/2008	22.5	480	8.33	44.6	8.70
5/25/2008	22.5	480	8.33	50.8	8.70
5/25/2008	22.5	480	8.33	41.0	8.70
5/25/2008	22.5	480	8.33	41.6	8.70
5/25/2008	22.5	480	8.33	37.9	8.69
5/25/2008	22.5	480	8.33	41.0	8.70
5/25/2008	22.5	480	8.33	39.1	8.70
5/25/2008	22.5	481	8.33	47.2	8.70
5/25/2008	22.5	481	8.33	37.2	8.69
5/25/2008	22.5	481	8.33	40.3	8.70
5/25/2008	22.5	481	8.33	44.3	8.69
5/26/2008	22.5	481	8.33	39.7	8.70
5/26/2008	22.5	481	8.33	41.2	8.70
5/26/2008	22.5	481	8.33	40.8	8.70
5/26/2008	22.5	481	8.33	38.8	8.70
5/26/2008	22.5	481	8.33	46.0	8.69
5/26/2008	22.4	481	8.33	40.2	8.70
5/26/2008	22.4	481	8.33	40.5	8.70
5/26/2008	22.4	481	8.32	36.8	8.71
5/26/2008	22.4	481	8.32	41.9	8.71
5/26/2008	22.4	481	8.32	46.2	8.70
5/26/2008	22.4	482	8.32	42.9	8.71
5/26/2008	22.4	482	8.32	45.2	8.71
5/26/2008	22.4	482	8.32	39.5	8.72
5/26/2008	22.4	482	8.32	39.1	8.71
5/26/2008	22.4	482	8.32	36.8	8.71
5/26/2008	22.4	482	8.32	42.6	8.71
5/26/2008	22.4	482	8.32	43.6	8.72
5/26/2008	22.4	482	8.32	38.2	8.72

5/26/2008	22.4	482	8.32	41.1	8.72
5/26/2008	22.4	482	8.32	39.6	8.72
5/26/2008	22.3	482	8.32	38.0	8.72
5/26/2008	22.3	482	8.32	39.7	8.73
5/26/2008	22.3	482	8.32	41.1	8.73
5/26/2008	22.3	482	8.32	41.0	8.73
5/26/2008	22.3	482	8.32	34.9	8.73
5/26/2008	22.3	482	8.32	38.3	8.73
5/26/2008	22.3	483	8.32	41.0	8.73
5/26/2008	22.4	483	8.32	37.4	8.73
5/26/2008	22.4	483	8.32	35.5	8.72
5/26/2008	22.4	483	8.32	37.4	8.72
5/26/2008	22.4	483	8.32	39.7	8.72
5/26/2008	22.4	483	8.32	38.2	8.72
5/26/2008	22.4	483	8.32	41.6	8.72
5/26/2008	22.4	483	8.32	39.6	8.72
5/26/2008	22.5	483	8.32	36.4	8.71
5/26/2008	22.5	483	8.32	43.9	8.71
5/26/2008	22.5	483	8.32	42.2	8.71
5/26/2008	22.5	483	8.32	44.6	8.71
5/26/2008	22.5	484	8.32	37.0	8.71
5/26/2008	22.5	484	8.32	38.0	8.71
5/26/2008	22.5	484	8.32	35.1	8.70
5/26/2008	22.6	484	8.32	38.5	8.70
5/26/2008	22.6	484	8.32	38.3	8.70
5/26/2008	22.6	484	8.32	39.6	8.70
5/26/2008	22.6	484	8.32	35.0	8.69
5/26/2008	22.6	484	8.32	38.1	8.69
5/26/2008	22.6	484	8.32	38.8	8.69

5/26/2008	22.7	485	8.32	39.8	8.68
5/26/2008	22.7	485	8.33	34.9	8.68
5/26/2008	22.7	485	8.33	37.2	8.68
5/26/2008	22.7	485	8.33	36.4	8.68
5/26/2008	22.7	485	8.33	34.4	8.68
5/26/2008	22.7	485	8.33	33.5	8.68
5/26/2008	22.7	485	8.33	35.7	8.68
5/26/2008	22.7	485	8.33	35.6	8.68
5/26/2008	22.7	485	8.33	34.9	8.68
5/26/2008	22.8	485	8.33	38.2	8.68
5/26/2008	22.8	485	8.33	32.6	8.67
5/26/2008	22.8	485	8.34	35.0	8.67
5/26/2008	22.8	486	8.34	32.9	8.67
5/26/2008	22.8	486	8.34	34.3	8.66
5/26/2008	22.8	486	8.34	33.1	8.66
5/26/2008	22.8	486	8.34	35.3	8.66
5/26/2008	22.8	486	8.34	36.1	8.66
5/26/2008	22.8	486	8.34	32.8	8.66
5/26/2008	22.8	486	8.34	33.8	8.66
5/26/2008	22.8	486	8.34	32.7	8.66
5/26/2008	22.8	486	8.34	35.7	8.66
5/26/2008	22.8	486	8.34	32.3	8.66
5/26/2008	22.8	486	8.34	32.8	8.65
5/26/2008	22.8	486	8.34	32.2	8.66
5/26/2008	22.8	487	8.34	31.6	8.66
5/26/2008	22.9	486	8.34	30.3	8.66
5/26/2008	22.8	487	8.34	34.6	8.66
5/26/2008	22.9	487	8.34	30.5	8.66
5/26/2008	22.9	487	8.35	34.1	8.66

5/26/2008	22.9	487	8.35	32.1	8.65
5/26/2008	22.9	487	8.35	31.8	8.66
5/26/2008	22.9	487	8.35	31.4	8.66
5/26/2008	22.9	487	8.35	32.7	8.66
5/26/2008	22.9	487	8.35	31.4	8.66
5/26/2008	22.9	487	8.35	33.0	8.66
5/26/2008	22.9	487	8.35	30.4	8.66
5/26/2008	22.9	487	8.35	30.8	8.66
5/26/2008	22.9	487	8.35	31.6	8.66
5/26/2008	22.9	488	8.35	29.4	8.66
5/26/2008	22.9	488	8.35	30.6	8.66
5/26/2008	22.9	488	8.35	30.8	8.66
5/26/2008	22.9	488	8.35	30.2	8.66
5/26/2008	22.9	488	8.35	31.6	8.66
5/26/2008	22.9	488	8.35	32.2	8.66
5/26/2008	22.9	488	8.35	31.4	8.67
5/26/2008	22.8	488	8.35	29.2	8.67
5/26/2008	22.8	488	8.35	29.9	8.67
5/26/2008	22.8	488	8.35	27.8	8.68
5/26/2008	22.8	488	8.35	27.8	8.68
5/27/2008	22.8	488	8.35	33.8	8.68
5/27/2008	22.8	488	8.35	31.4	8.68
5/27/2008	22.8	488	8.35	30.8	8.68
5/27/2008	22.8	488	8.35	31.4	8.69
5/27/2008	22.7	489	8.35	28.0	8.69
5/27/2008	22.7	489	8.35	27.8	8.69
5/27/2008	22.7	489	8.35	30.0	8.69
5/27/2008	22.7	489	8.35	30.0	8.69
5/27/2008	22.7	489	8.35	30.0	8.69

5/27/2008	22.7	489	8.35	29.0	8.69
5/27/2008	22.7	489	8.35	28.6	8.70
5/27/2008	22.7	489	8.35	29.9	8.69
5/27/2008	22.7	489	8.35	28.3	8.70
5/27/2008	22.7	489	8.35	26.9	8.70
5/27/2008	22.7	489	8.35	26.3	8.70
5/27/2008	22.7	489	8.34	28.9	8.70
5/27/2008	22.7	489	8.35	27.4	8.70
5/27/2008	22.7	489	8.35	26.3	8.71
5/27/2008	22.7	490	8.35	26.1	8.71
5/27/2008	22.7	490	8.35	26.7	8.71
5/27/2008	22.7	490	8.35	25.5	8.71
5/27/2008	22.7	490	8.35	26.7	8.71
5/27/2008	22.7	490	8.35	26.2	8.71
5/27/2008	22.7	490	8.35	26.4	8.71
5/27/2008	22.7	490	8.35	25.8	8.72
5/27/2008	22.7	490	8.35	27.6	8.71
5/27/2008	22.7	490	8.35	26.5	8.71
5/27/2008	22.7	490	8.35	29.0	8.71
5/27/2008	22.7	490	8.35	25.8	8.71
5/27/2008	22.8	490	8.35	24.5	8.71
5/27/2008	22.8	490	8.35	24.7	8.71
5/27/2008	22.8	490	8.35	24.9	8.71
5/27/2008	22.8	491	8.35	25.3	8.70

Ni-SPIKED KAOLINITE (50 NTU) SRF EXPERIMENT 25-27 MAY 2008

Statistic	Temperature (°C)	Specific Conductance ($\mu S\ cm^{-1}$)	pH (units)	Turbidity (NTU)	DO ($mg\ L^{-1}$)
Mean	22.4	483	8.33	38.4	8.74
±SD	0.5	4	0.02	8.3	0.10
Minimum	20.7	475	8.26	24.5	8.65
Maximum	22.9	491	8.35	61.1	9.08

Hardness ($mg\ L^{-1}$ as $CaCO_3$)	Alkalinity ($mg\ L^{-1}$ as $CaCO_3$)
99	92

Appendix E: Grain size of Warden Ditch sediment

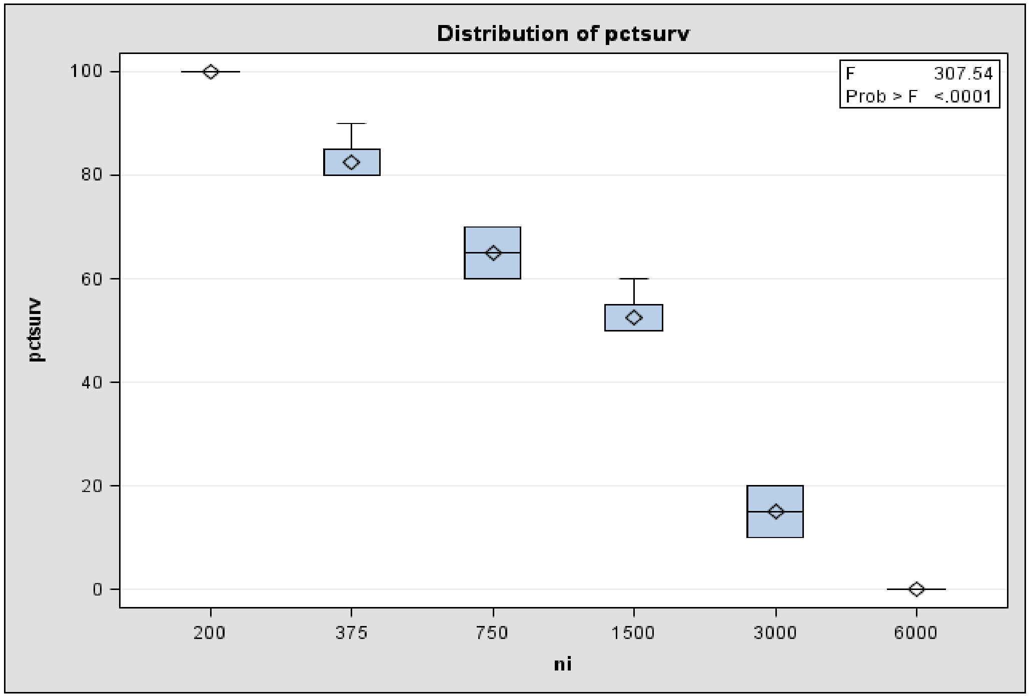
Appendix table 8. Determination of percentage of clay in WD sediment

Appendix table 8. Determination of percentage of clay in WD sediment

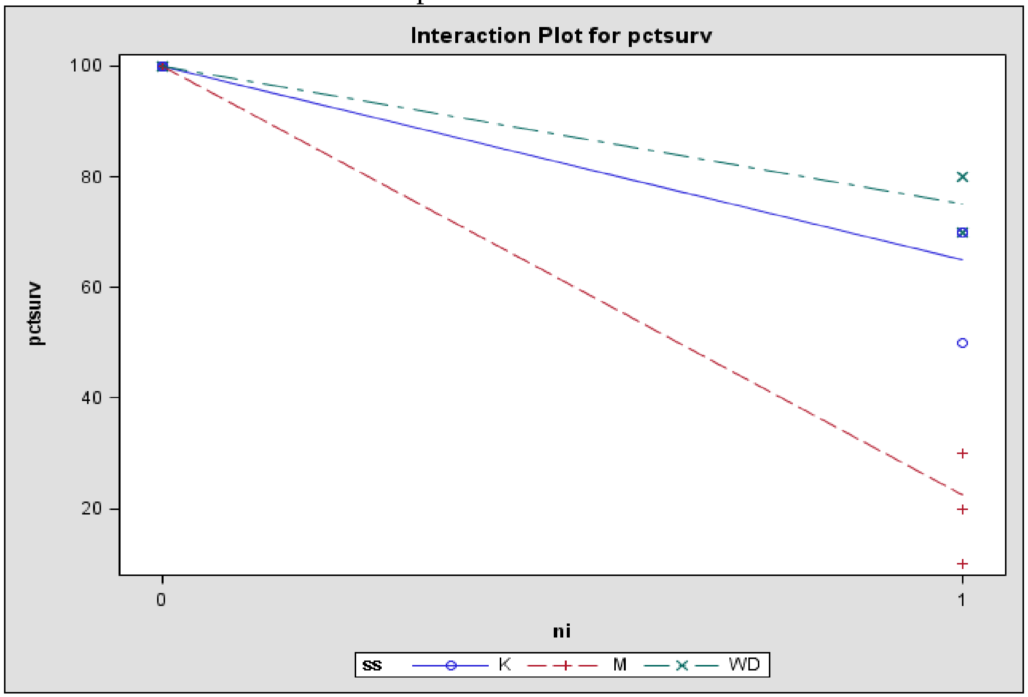
DETERMINING AMOUNT OF CLAY IN WARDEN DITCH SEDIMENT											
Replicate	**original sediment sample mass (g)**	**beaker (for silt) weight (g)**	**beaker + silt weight (g)**	**silt weight (g)**	**% silt**	**pan weight (g)**	**pan + sand weight (g)**	**sand weight (g)**	**% sand**	**% clay (100-[silt+sand])**	**Mean % clay**
1	30.2299	218.04	222.01	3.97	13.13	4.1927	7.9804	3.7877	12.53	74.34	74.93
2	30.191	215.45	219.09	3.64	12.06	4.1153	11.7555	3.7402	12.39	75.55	
3	30.2129	220.76	224.63	3.87	12.81	4.1378	6.8601	3.7623	12.45	74.74	
4	30.1733	221.3	225.11	3.81	12.63	4.1201	7.8211	3.701	12.27	75.11	

Appendix F: Statistical plots and tables

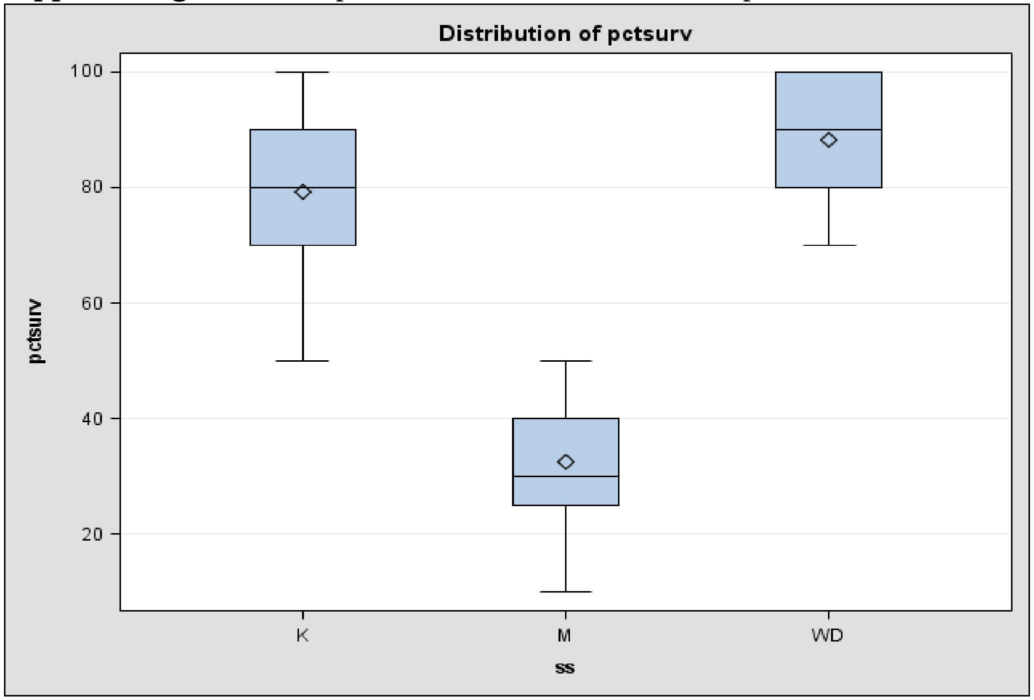
Appendix figure 1. Box plot of the distribution of mean percent survival versus total nickel concentration



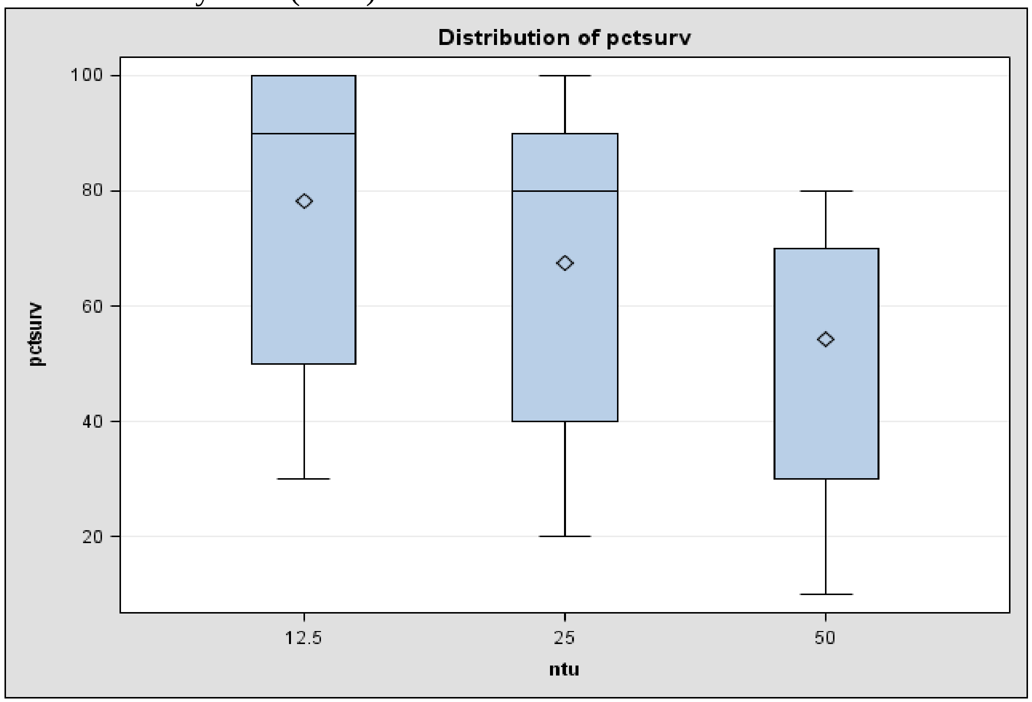
Appendix figure 2. Interaction plot showing mean percent survival versus SS with and without the presence of Ni



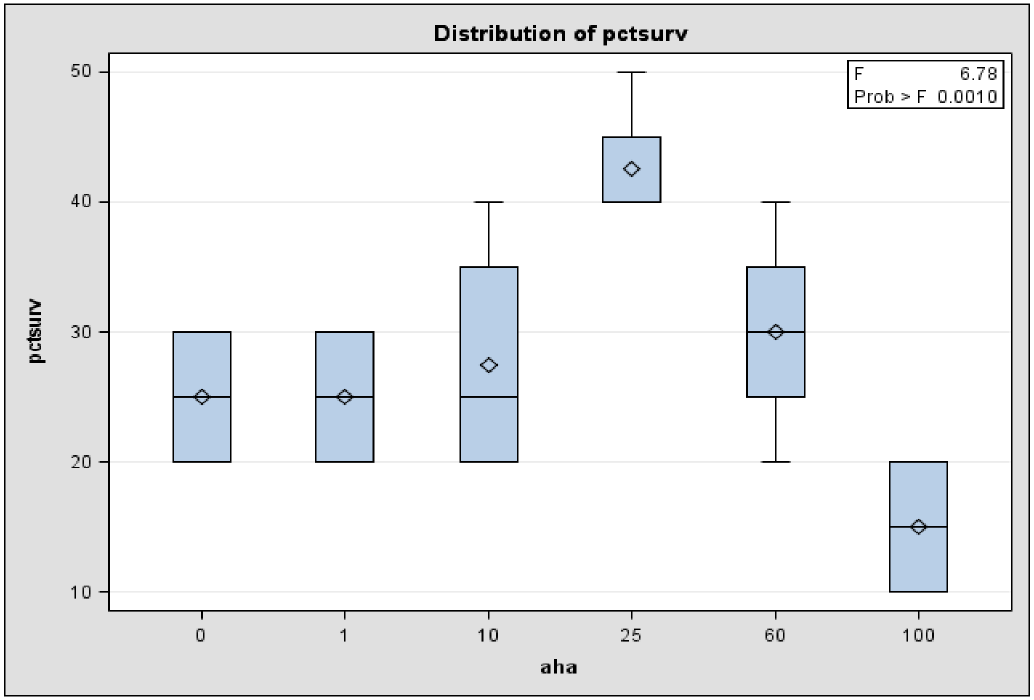
Appendix figure 3. Box plot of the distribution of mean percent survival versus SS



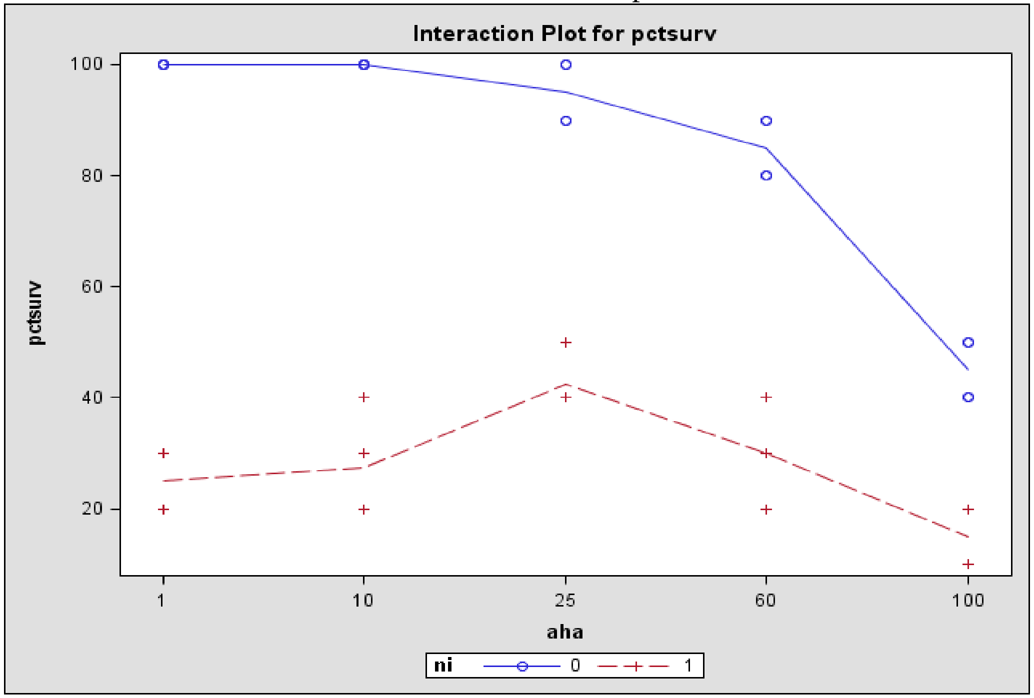
Appendix figure 4. Box plot of the distribution of mean percent survival versus turbidity level (NTU)



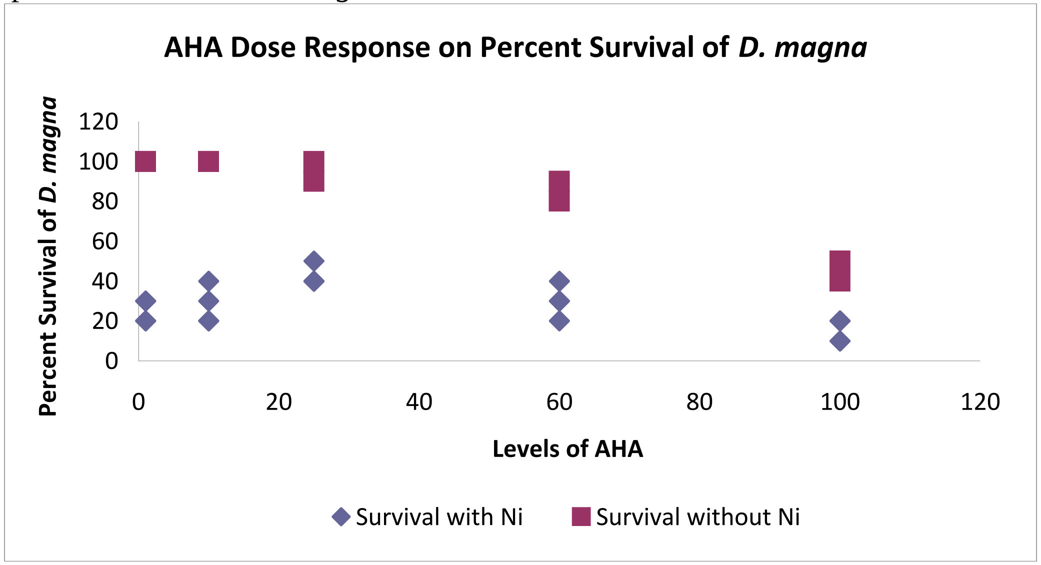
Appendix figure 5. Box plot of the distribution of mean percent survival versus AHA concentration



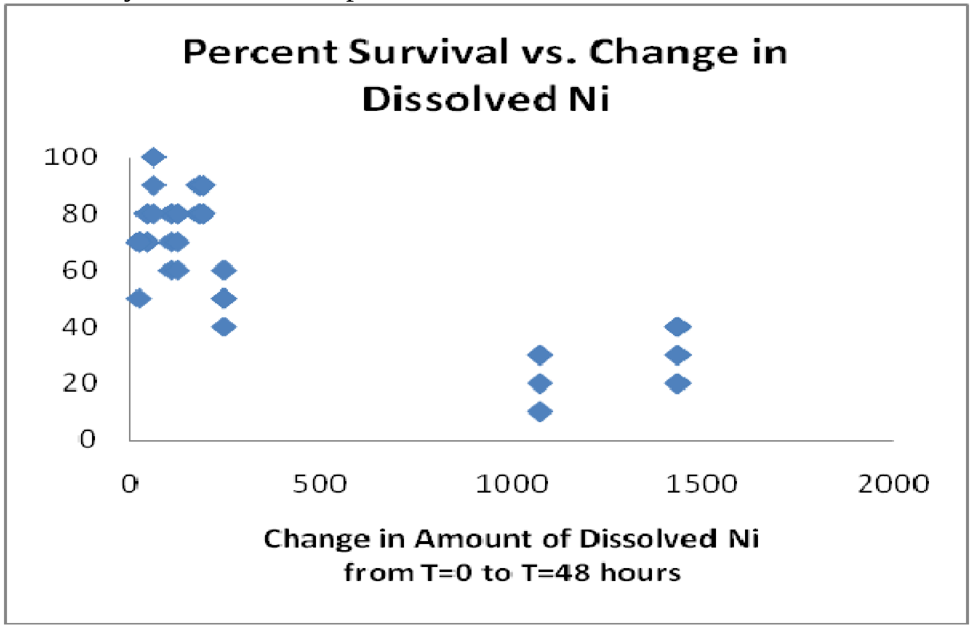
Appendix figure 6. Interaction plot showing mean percent survival versus AHA concentration with and without the presence of Ni



Appendix figure 7. Scatter plot of the dose response of AHA on mean percent survival of *D. magna*



Appendix figure 8. Dissolved Ni over 48 h: Significant predictor of the variability in the outcome percent survival



Appendix table 9. Concentrations of AHA that are significantly different with and without the presence of Ni

Concentrations of AHA ($mg\ L^{-1}$) that are significantly different (*)		Without Nickel					With Nickel				
		1	10	25	60	100	1	10	25	60	100
Without Nickel	1				*	*	*	*	*	*	*
	10				*	*	*	*	*	*	*
	25					*	*	*	*	*	*
	60	*	*			*	*	*	*	*	*
	100	*	*	*	*		*	*		*	*
With Nickel	1	*	*	*	*	*			*		
	10	*	*	*	*	*			*		
	25	*	*	*	*		*	*			*
	60	*	*	*	*	*					*
	100	*	*	*	*	*			*	*	

Appendix table 10. Change in dissolved Ni over 48 h among four arenas: Observations, their ranks, and marginal means

Change in dissolved Ni (Dis0 – Dis48) (rank)	WD	Montmorillonite	Kaolinite	Arena Means [SD]
SRF Channel SS + Ni	190 (8)	1433 (11)	123 (6)	582.0 [737.7]
SRF Chamber SS + Ni	179 (7)	1438 (12)	106 (5)	574.3 [748.8]
Batch SS + Ni	43 (2)	1074 (10)	22 (1)	379.7 [601.4]
Batch SS + Ni + AHA	60 (4)	245 (9)	46.7 (3)	117.2 [110.9]
SS Means [SD]	118.0 [77.2]	1047.5 [561.5]	74.4 [47.9]	

Appendix table 11. Change in sorbed Ni over 48 h among four arenas: Observations, their ranks, and marginal means

Change in sorbed Ni (Sorb48 – Sorb0) (rank)	WD	Montmorillonite	Kaolinite	Arena Means [SD]
SRF Channel SS + Ni	-69 (2)	256 (8)	-3 (5)	61.3 [171.8]
SRF Chamber SS + Ni	1500 (12)	1070 (11)	409 (10)	993.0 [549.6]
Batch SS + Ni	-75 (1)	308 (9)	2 (6)	78.3 [202.6]
Batch SS + Ni + AHA	-63 (3)	-21 (4)	140 (7)	18.7 [107.2]
SS Means [SD]	323.3 [784.5]	403.3 [467.4]	137.0 [193.1]	